

UNCLASSIFIED

AD 491754

DEFENSE DOCUMENTATION CENTER

FOR

SCIENTIFIC AND TECHNICAL INFORMATION

CAMERON STATION ALEXANDRIA, VIRGINIA



UNCLASSIFIED

NOTICE: When government or other drawings, specifications or other data are used for any purpose other than in connection with a definitely related government procurement operation, the U. S. Government thereby incurs no responsibility, nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use or sell any patented invention that may in any way be related thereto.

(11) 18 Feb 1941,

(14) NRL-H-1692

UNANNOUNCED

NAVY DEPARTMENT

(6) ~~SECRET~~
Metallic Fluoride Films
of Low Reflectivity.

NAVAL RESEARCH LABORATORY
ANACOSTIA STATION
WASHINGTON, D.C.

(12) 60 p.

Number of Pages: Text ~~1~~ Tables ~~3~~ Plates ~~12~~

Authorization: BuShips Project Order No. 135/41 of 28 July 1940

Rept. for
1 July 1940 to 13 January 1941

Prepared by: (10) J. A. Sanderson, Assistant Physicist, X-10 E

Reviewed by: E. O. Hulbert, Principal Physicist, Superintendent, Physical Optics Division.

Approved by: H. G. Bowen, Rear Admiral, USN, Director.

Distribution: BuShips (5)

cjh

Distribution of this document is unlimited.

Best Available Copy

**Best
Available
Copy**

Table of Contents

<u>Subject</u>	<u>Page</u>
Abstract	
Authorization	1
Chapter I.	
Introduction	1
Description of the Films	1
Evaporated Films: Summary of Known Facts	1
Qualitative Examples	2
Chapter II.	
Reflection of Light by Optical Surfaces	4
Reflection by Glass	4
Thin Film Interference	6
Relation Between Refractive Indexes of Glass and Film Material	7
Effect of Film Thickness	8
Highly Reflecting Films	10
Chapter III.	
Optical Properties: Experimental Results	12
Binoculars	12
Transmission Measurements	13
Effects of Staggered Film Thicknesses	15
Water-Proofing	16
Index of Refraction of the Films	17
Chapter IV.	
Vacuum System	20
Diffusion Pumps	20
Oil Diffusion Pumps	21
Choice of Diffusion Pump	22
Fore Pumps	22
The Vacuum Chamber	23
Valves and Gaskets	24
Flatness of the Base Plate	25
Filaments	25
Transformers	26
Vacuum Gauges	26
Conclusion	27
Chapter V.	
The Evaporation Process	28
Cleaning the Glass	28
Removal of Lint and Dust	28
Testing for Cleanliness	29
Cleaning with Electric Discharge	29
Preliminary Treatment of Glass	30
Evaporation of the Films	30
Determining the Film Thickness	31
Physiological Note	32
Chapter VI.	
Mechanical Properties of the Films	33
Hardness	33
Hardening by Heat	33
Hardening with Age	33
Effect of Water	33

Table of Contents (Continued)

<u>Subject</u>	<u>Page</u>
Chapter VII.	
Summary and Conclusions	35
Diffusion Pumps	35
Mechanical Pumps	35
Valves and Gaskets	35
Pressure Gauges	36
Film Materials	36
New Research	36

Appendices

Transmission by Glass Plates	Table I
Transmission Factors of 7x50 Binoculars Coated with Metallic Fluoride Films	II
Properties of Metallic Fluorides	III
Appendix A - Sources of Supply	Page -a-

References

Photograph through Two Glass Plates Coated with MgF_2 Over the Central Areas	Plate 1
Photographs of 500 Watt Light with Zeiss Planar Lens, 8 Surfaces. Left: Uncoated; Right: Coated with CaF_2 . .	2
Phase Relation Between Waves Reflected from the Front and Back Surfaces of a Thin Film	3
Transmission by MgF_2 Films	4
Transmission by MgF_2 Films	5
Transmission by CaF_2 Films	6
Transmission by 10 Glass Plates: (a) 10 Red, 10 Blue Films; (b) All Films Purple	7
Transmission by Cryolite Films (10 Surfaces)	8
Transmission of MgF_2 Films Before and After Waxing	9
Diffusion Pump	10
Vacuum Chamber	11
Pumping Speeds of Mechanical Pumps	12

FEB 27 1941

ABSTRACT

The method of reducing the reflection losses at glass surfaces by depositing thin films of magnesium fluoride, calcium fluoride, or sodium aluminum fluoride on the glass by evaporation in high vacuum, as perfected by Cartwright and Turner, has been investigated in detail. The action of the films depends on two things: first, their refractive indexes are much lower than the refractive index of glass so that even a thin layer of the film material reduces the reflection; and second, when the films are made to be one-quarter wave-length thick for any chosen wave-length, a minimum of reflected light intensity results from interference of the waves reflected from the front and back surfaces of the film. The effect of film thickness is illustrated by a calculation of the transmission for monochromatic light of a glass plate of refractive index 1.65 coated on both sides with cryolite films assumed to have a refractive index of 1.35, a reasonable value. The results of the calculations are:

Transmission of bare glass	88.4 per cent
Transmission of glass coated with very thin film (no interference)	93.7 per cent
Transmission of glass coated with films of optical thickness one-fourth wave-length	99.6 per cent

The films are most effective when their optical thickness is one-fourth the wave-length of green light, about 5500 AU, in the middle of the visible spectrum where the eye has maximum sensitivity. Films of this thickness reflect red and blue light more strongly than green and so appear purple when examined in reflected light. The effect of reducing the reflection by the glass is to increase the transmission; the transmission of white light by a single crown glass surface may be increased from 96 per cent to 99 per cent by a purple magnesium fluoride film. The spectral transmission curve of such a coated surface, deduced from measurements on several plates coated with the films, may be 98.1 per cent at 4000 AU, 99.2 per cent at 5500 AU, and 98.2 per cent at 6000 AU. When the white light transmission is measured with the eye as receiver, the value obtained is usually about equal to the spectral transmission near the middle of the spectrum where the (purple) film has highest transmission, because the eye sensitivity is low in the blue and the red.

Films of magnesium fluoride and sodium aluminum fluoride (cryolite) are hard enough to withstand the handling necessary for the assembly of instruments, when the evaporation takes place at pressures lower than 10^{-4} mm Hg; films of calcium fluoride are soft and easily wiped off. Magnesium fluoride films can be further hardened by heating to 400°C. This is not necessary except for external optical parts subjected to frequent cleaning; it is perhaps better to leave such surfaces uncoated. Magnesium fluoride films are harder than cryolite films; either should be satisfactory for protected optical parts. Immersion in water for several days does not

(A)

impair the optical properties of the films although it softens them. They can be made water proof by waxing but with serious impairment of the optical properties.

The evaporation process and suitable pumping systems for producing the requisite high vacua are discussed in detail.

I. AUTHORIZATION

1. Work on this problem was authorized by Bureau of Ships Project Order No. 135/41 of 26 July 1940.

Chapter I.

INTRODUCTION

Description of the Films

2. This report describes the properties of thin films of metallic fluorides which, when deposited in the proper thicknesses on glass surfaces by the method of evaporation in high vacuum, reduce appreciably the losses of light due to reflection and, therefore, increase the transmission. Another effect of the diminution of reflected light is to decrease the intensity of field light with resultant improved contrast and effective resolving power in both visual and photographic instruments.

3. While the optical phenomena upon which the action of the films depends and the method by which they are prepared have been long familiar, it remained for Cartwright and Turner(1) to achieve practicability for the process by demonstrating that optical instruments so treated withstand ordinary usage and show greatly improved performance over similar untreated instruments. Much of the information included in this report was obtained during the period of a month that Dr. Cartwright spent in residence at this Laboratory.

4. The historical development of methods for increasing the transmission of optical surfaces has been outlined by Cartwright and Turner(1), Turner(2), and Blodgett(3). While at present the evaporated film method is optically the most effective practical means of achieving the result, advances continue to be made in chemical treatment of glass surfaces. A recent abstract by Jones and Homer(4) describes a method of treating freshly prepared glass surfaces with dilute acid solutions which "... involves the formation of a transparent surface film of silica by the removal of the higher refractive index oxides to a depth approximately equal to one-fourth the wave-length of the light for which maximum transmission is desired. . . . The surface film is not noticeably different from the base glass in hardness. The gain in light transmission and the decrease in surface reflection is slightly less than that produced by evaporated films of materials of lower refractive index than silica."

Evaporated Films: Summary of Known Facts

5. This report deals only with evaporated films of magnesium fluoride, calcium fluoride, and sodium aluminum fluoride (cryolite) for reducing reflection, and zinc sulphide for increasing the efficiency of half reflecting mirrors. Facts that have been determined experimentally are summarized below.

- (a) Evaporated films of the fluorides one-quarter wave-length of green light thick increase the over all transmission of a single crown glass surface for visible light from 96

per cent to about 99 per cent. These figures represent a decrease in reflection loss of 75 per cent.

- b) While all the compounds named are suitable for use on protected optical parts, magnesium fluoride forms the hardest and most durable films, and these films can be further hardened by heating to 400°C.
- c) Cryolite films are moderately hard when deposited at low pressures but cannot be made harder by heating.
- d) Both magnesium fluoride and cryolite films are sufficiently hard to withstand gentle rubbing with cloth or lens paper and the handling necessary for assembly of instruments.
- e) Calcium fluoride films are soft but of good optical properties because the refractive index of the soft films seems to be low.
- f) The production of hard films of magnesium fluoride or cryolite requires that the evaporation be carried out at pressures less than 10^{-4} mm Hg.
- g) The films can be waterproofed and made more resistant to abrasion by oiling or waxing, but their optical properties, while superior to bare glass surfaces, are inferior to unwaxed films.
- h) The solubilities of the films in water are low. Soaking the films in water does not remove them but only softens them.
- i) The freshly prepared films are good adsorbers and condense moisture more readily than bare glass. In aging they acquire a monomolecular film of oil from the atmosphere which offers some protection against moisture without impairing their efficiency.

Qualitative Examples

6. The effect of the films in reducing the reflection of light by glass surfaces is qualitatively shown by Plate 1 which is a photograph through two plates of glass, each coated on both sides over the circular central area with magnesium fluoride. In making this photograph the plates were so oriented that bright daylight from a window was directly reflected into the camera, and while the intensity of the light reflected by the uncoated edges of the plates was so great that the underlying print is barely legible in the picture, the intensity was not greatly reduced in passing through the low reflecting area. This photograph through two glass plates represents transmission by four plates since the light reaching the camera traversed the plates twice. The transmission of white light by four such coated plates is usually observed to be about 87 per cent and of four uncoated surfaces, about 71 per cent. These figures mean that a single surface of the untreated glass reflects 4 per cent of the light while the coated surface reflects 1 per cent.

7. Plate 2 shows photographs of a 500 watt projection lamp made on panchromatic film with two identical Zeiss Planar f:4 lenses, that used for the right hand picture being coated with calcium fluoride, and the other uncoated. Each lens contained eight glass-air surfaces. Multiple reflections in the uncoated lens produced the intense flare rings seen in the left hand photograph and gave rise to strong field light over the whole photograph, but the reflected intensities were so low that the flare image is barely perceptible in the right hand photograph, the sharpness with which the glass envelope of the lamp is delineated is much improved, and the scattered light over the field is less intense. In many photographic instruments the gain in contrast is more important than the increase in light gathering power that the films bring about. The relative light gathering power of the two lenses cannot be estimated from the photographs, but measurements of relative transmission showed that of the coated lens to be 1.34 times that of the uncoated lens.

8. Other illustrative examples are given in the paper by Turner⁽²⁾.

Chapter II.

REFLECTION OF LIGHT BY OPTICAL SURFACES

Reflection by Glass

9. When light is incident upon a boundary between two transparent materials of different refractive indexes, as the interface between air and glass, a part of it is reflected. The amplitudes of the reflected waves are given for any angle of incidence by Fresnel's equations,

$$\begin{array}{l} \text{Reflected} \\ \text{amplitudes} \end{array} \quad \left\{ \begin{array}{l} A_s = -E_s \frac{\sin(i-r)}{\sin(i+r)} \\ A_p = E_p \frac{\tan(i-r)}{\tan(i+r)} \end{array} \right. \quad (1)$$

where subscripts s and p refer to the components of the electric vector of magnitude E perpendicular and parallel to the plane of incidence, respectively, i is the angle of incidence and r is the angle of refraction. In the special case which will be of greatest interest, $i = r = 0$ and the expressions are indeterminate since $\sin(i-r) = \sin(i+r) = 0$ and $\tan(i-r) = \tan(i+r) = 0$.

In the limit, however, as $i \rightarrow r \rightarrow 0$, $\sin(i-r) \rightarrow (i-r)$, $\sin(i+r) \rightarrow (i+r)$, etc. and, putting $i = rn$ where n is the index of refraction of the medium,

$$\begin{aligned} A_s &= -E_s \left(\frac{i-r}{i+r} \right) = -E_s \left(\frac{n-1}{n+1} \right) \\ A_p &= E_p \left(\frac{i-r}{i+r} \right) = E_p \left(\frac{n-1}{n+1} \right) \end{aligned} \quad (2)$$

The intensity of the reflected light is given by the square of equations (2),

$$\begin{aligned} R_s &= E_s^2 \left(\frac{n-1}{n+1} \right)^2, \\ R_p &= E_p^2 \left(\frac{n-1}{n+1} \right)^2, \\ \text{or } R &= I \left(\frac{n-1}{n+1} \right)^2 \end{aligned} \quad (3)$$

where $I = E^2$ is the intensity of the incident light.

10. If $I = 1$, then R is the reflectivity of the surface, and for crown glass of refractive index $n = 1.5$ it readily results from (3) that

$$R = 0.04 ,$$

$$T = (1 - R) = 0.96 ,$$

for a single glass surface; or for a set of plates or lenses presenting N surfaces,

$$T_N = (1 - R)^N , \quad (4)$$

neglecting absorption within the glass.

11. Table I gives values of T_N for several values of N for three kinds of glass and, in column 5, the transmission by surfaces reflecting only 1 per cent of the incident light. This is about the value to which low reflecting films reduce the overall white light reflectivity of crown glass. The table is inserted merely for convenience in forming an estimate of the improvement to be expected by coating an instrument of N surfaces with fluoride films.

12. Equation (3) gives the reflection by a glass surface in contact with air. If two optical surfaces of any refractive indexes n_1 and n_2 are in contact, the reflectivity is given by

$$R = \left(\frac{n_1 - n_2}{n_1 + n_2} \right)^2 \quad (5)$$

which shows that if glass is covered with a layer of any material of lower refractive index, as water, the reflection by the water-glass interface is considerably reduced and the total intensity of the reflected light, made up of the part reflected by the air-water surface and that reflected from the water-glass surface, is less than the intensity of the light reflected by a single bare glass surface. If

$$n_{\text{glass}} = 1.5 \text{ and } n_{\text{water}} = 1.33, \text{ then}$$

$$R_{\text{water}} = \left(\frac{1.33 - 1}{1.33 + 1} \right)^2 = 0.0196 ,$$

$$R_{\text{water-glass interface}} = \left(\frac{1.5 - 1.33}{1.5 + 1.33} \right)^2 = 0.0036 .$$

If light of unit intensity falls on glass covered with water, the transmission factor is

$$T = (1 - R_w)(1 - R_{wg}) = 0.977$$

which is greater than the transmission by a single bare glass surface.

10. If $I = 1$, then R is the reflectivity of the surface, and for crown glass of refractive index $n = 1.5$ it readily results from (3) that

$$R = 0.04 ,$$

$$T = (1 - R) = 0.96 ,$$

for a single glass surface; or for a set of plates or lenses presenting N surfaces,

$$T_n = (1 - R)^N , \quad (4)$$

neglecting absorption within the glass.

11. Table I gives values of T_N for several values of N for three kinds of glass and, in column 5, the transmission by surfaces reflecting only 1 per cent of the incident light. This is about the value to which low reflecting films reduce the overall white light reflectivity of crown glass. The table is inserted merely for convenience in forming an estimate of the improvement to be expected by coating an instrument of N surfaces with fluoride films.

12. Equation (3) gives the reflection by a glass surface in contact with air. If two optical surfaces of any refractive indexes n_1 and n_2 are in contact, the reflectivity is given by

$$R = \left(\frac{n_1 - n_2}{n_1 + n_2} \right)^2 \quad (5)$$

which shows that if glass is covered with a layer of any material of lower refractive index, as water, the reflection by the water-glass interface is considerably reduced and the total intensity of the reflected light, made up of the part reflected by the air-water surface and that reflected from the water-glass surface, is less than the intensity of the light reflected by a single bare glass surface. If

$$n_{\text{glass}} = 1.5 \text{ and } n_{\text{water}} = 1.33, \text{ then}$$

$$R_{\text{water}} = \left(\frac{1.33 - 1}{1.33 + 1} \right)^2 = 0.0196 ,$$

$$R_{\text{water-glass interface}} = \left(\frac{1.5 - 1.33}{1.5 + 1.33} \right)^2 = 0.0036 .$$

If light of unit intensity falls on glass covered with water, the transmission factor is

$$T = (1 - R_w)(1 - R_{wg}) = 0.977$$

is greater than the transmission by a single bare glass surface.

13. This, however, is not the complete description of the action of metallic fluoride films in reducing the reflection. A more complete discussion follows.

Thin Film Interference

14. The reduction of reflection losses at glass surfaces is achieved in the method of Cartwright and Turner by depositing on the glass a thin film of a metallic fluoride (chosen because of their low refractive indexes and their mechanical properties) of such thickness that the wave trains reflected from the front (air to film) and back (film to glass) surfaces have a phase difference of one-half wave-length and interact to give a minimum of reflected light intensity by interference. The reflected intensity is even less than that obtained by covering the glass with a very thick (or thin) film of lower refractive index as in paragraph 12.

15. Plate 3 represents a plate of glass G, refractive index n_g , covered by a film F of refractive index n_f and thickness t . A beam of light, AB, incident on the film will be in part reflected along BH and in part transmitted along BC. BC in turn is in part reflected along CE and EG and in part transmitted into the glass along CA'. It is necessary to determine the conditions under which the two reflected wave trains BH and EG will combine to give a minimum of reflected light intensity by interference for light of any wave-length λ , which is to say that the conditions under which BH and EG differ in phase by $1/2 \lambda$ must be known.

16. It is a consequence of equations (1) - (2) that since $n_f > 1$, AB undergoes a phase change of $1/2 \lambda$ upon reflection from the film, and, if $n_g > n_f$, BC suffers a phase change of $1/2 \lambda$ upon reflection at the film-glass interface, so that the process of reflection introduces no phase difference. However, the emergent wave travelling along EG has traversed a distance $(BC + CE) - BF$ greater than that travelled by BH, and a part of this path lies in the film material of refractive index n_f , so that the additional path travelled by EG is equivalent to an airpath of length $n_f (BC + CE) - BF$. It is shown on Plate 3 that the retardation of EG over BH is

$$\Delta_{EG-BH} = 2n_f t \cos i, \quad (6)$$

where i is the angle of incidence at the film-glass surface and t is the film thickness. Since for a minimum of reflected light intensity by interference

$$\Delta = 1/2 \lambda,$$

it follows that the film thickness must be such that

$$\begin{aligned} 2 n_f t \cos i &= 1/2 \lambda, \\ n_f t \cos i &= 1/4 \lambda. \end{aligned} \quad (7)$$

13. This, however, is not the complete description of the action of metallic fluoride films in reducing the reflection. A more complete discussion follows.

Thin Film Interference

14. The reduction of reflection losses at glass surfaces is achieved in the method of Cartwright and Turner by depositing on the glass a thin film of a metallic fluoride (chosen because of their low refractive indexes and their mechanical properties) of such thickness that the wave trains reflected from the front (air to film) and back (film to glass) surfaces have a phase difference of one-half wave-length and interact to give a minimum of reflected light intensity by interference. The reflected intensity is even less than that obtained by covering the glass with a very thick (or thin) film of lower refractive index as in paragraph 12.

15. Plate 3 represents a plate of glass G, refractive index n_g , covered by a film F of refractive index n_f and thickness t . A beam of light, AB, incident on the film will be in part reflected along BH and in part transmitted along BC. BC in turn is in part reflected along CE and EG and in part transmitted into the glass along CA'. It is necessary to determine the conditions under which the two reflected wave trains BH and EG will combine to give a minimum of reflected light intensity by interference for light of any wave-length λ , which is to say that the conditions under which BH and EG differ in phase by $1/2 \lambda$ must be known.

16. It is a consequence of equations (1) - (2) that since $n_g > 1$, AB undergoes a phase change of $1/2 \lambda$ upon reflection from the film, and, if $n_g > n_f$, BC suffers a phase change of $1/2 \lambda$ upon reflection at the film-glass interface, so that the process of reflection produces no phase difference. However, the emergent wave travelling along EG has traversed a distance $(BC + CE) - BF$ greater than that travelled by BH, and a part of this path lies in the film material of refractive index n_f , so that the additional path travelled by EG is equivalent to an airpath of length $n_f (BC + CE) - BF$. It is shown on Plate 3 that the retardation of EG over BH is

$$\Delta_{EG-BH} = 2n_f t \cos i, \quad (6)$$

where i is the angle of incidence at the film-glass surface and t is the film thickness. Since for a minimum of reflected light intensity interference

$$\Delta = 1/2 \lambda,$$

it follows that the film thickness must be such that

$$\begin{aligned} 2 n_f t \cos i &= 1/2 \lambda, \\ n_f t \cos i &= 1/4 \lambda. \end{aligned} \quad (7)$$

Usually, the light is incident normally so that $i = 0$, $\cos i = 1$, and the condition to be satisfied is

$$n_f t = 1/4 \lambda. \quad (8)$$

17. Equation (8) shows that to bring about a minimum of reflected light intensity for any wave-length λ , the product of the index of refraction and thickness of the film must be equal to one-quarter of the wave-length. Obviously, a film producing minimum reflection for one part of the spectrum will not be of the proper thickness for other wave-lengths. In practice the film thickness is made to satisfy (8) for λ about 5500 AU; it will be seen later that such a film is very effective over the visible spectrum.

Relation Between Refractive Indexes of Glass and Film Material

18. If the wave trains BH and EG are in opposite phase most complete interference will exist when, in addition, their amplitudes are equal.

The amplitude of BH is given by

$$A_{BH} = \frac{n_f - 1}{n_f + 1} \quad (9)$$

and of EG by

$$A_{EG} = A_{CE} = \frac{n_g - n_f}{n_g + n_f} \quad (10)$$

In this calculation the small correction for multiple reflection is neglected.

19. Thus, if $A_{BH} = A_{EG}$,

$$\frac{n_f - 1}{n_f + 1} = \frac{n_g - n_f}{n_g + n_f} \quad (11)$$

from which it turns out readily that the best value for the refractive index of the film is

$$n_f = \sqrt{n_g} \quad (12)$$

The ideal film material for use on crown glass would have a refractive index $n_f = 1.225$, and for denser glass $n_g = 1.65$, $n_f = 1.28$. The metallic fluorides used as film material have refractive indexes somewhat higher than these values; they are nevertheless the materials most nearly meeting the ideal conditions while at the same time having good mechanical properties.

Effect of Film Thickness

20. It was pointed out in paragraph 12 that as a consequence of equation (5) the reflectivity of a glass surface is reduced if it is covered with any transparent material of lower index of refraction, say, a thin (gray) film of cryolite. This has led French⁽⁵⁾ to the erroneous conclusion that, practically, there is no advantage to be derived from controlling the film thickness. That is (he concludes), so long as the glass has a fluoride film of any thickness on it the result should be satisfactory. In support of this contention he calculated the effect of cryolite films of refractive index $n_f = 1.35$ on dense glass, $n_g = 1.65$ without regard for phase. This is the same type of computation that was made in paragraph 12.

21. The reflectivity of the uncoated glass surface is -

$$R_G = \left(\frac{1.65 - 1}{1.65 + 1} \right)^2 = 0.06 ;$$

$$T_1 = 0.94 .$$

Transmission of the plate (two surfaces) is -

$$T_2 = (0.94)^2 = 0.884 .$$

Now the glass is coated with a film of refractive index 1.35 and no effect of interference is taken into consideration (as is allowable for a very thin film) the calculation of total reflected intensity is made in two steps:

Reflectivity of air-film surface

$$R_f = \left(\frac{1.35 - 1}{1.35 + 1} \right)^2 = 0.022 ;$$

$$T_f = 0.978 ;$$

Reflectivity of film-glass surface

$$R_{fg} = \left(\frac{1.65 - 1.35}{1.65 + 1.35} \right)^2 = 0.01 ;$$

$$T_{fg} = 0.99 .$$

Transmission of the glass plate coated on both sides with such a

$$T_2 = (0.978 \times 0.99)^2 = 0.937 .$$

The glass transmits 5.3 per cent more light than the uncoated glass. French states that this calculated gain is in accord with actual results in his institution*. It seems likely that his observations were

W. Stroud, Ltd., Glasgow.

made on films of proper thickness, for while the calculation is correct films too thin or too thick to show interference colors, in other words the film thickness profoundly affects the reflected light intensity, therefore, the transmission of the glass.

22. For example, if the effective film thickness is one-quarter wave-length so that equation (8) is satisfied for some wave-length λ , the waves reflected from the front and back surfaces of the film differ in phase by $1/2 \lambda$, and instead of adding intensities as in the calculation above, amplitudes must be added vectorially. If the intensity of the incident light is unity the amplitude of the wave reflected from the air-film surface is -

$$A_f = 1 \times \frac{1.35 - 1}{1.35 + 1} = 0.149 ,$$

$$R_f = (0.149)^2 = 0.022 .$$

The intensity reaching the film-glass interface is $(1 - 0.022)$ and the amplitude of the wave reflected at this interface is -

$$A_{fg} = (1 - 0.022) \left(\frac{1.65 - 1.35}{1.65 + 1.35} \right) = 0.098 .$$

The vector sum of these amplitudes is given by -

$$A^2 = R = A_f^2 + A_{fg}^2 + 2A_f A_{fg} \cos 2\pi \left(\frac{2n_f t}{\lambda} \right) \quad (13)$$

where $2\pi \left(\frac{2n_f t}{\lambda} \right)$ is the phase difference between the reflected waves,

in this case equal to π since

$$2n_f t = \frac{\lambda}{2} .$$

Substitution of the numerical values gives -

$$R = (0.149)^2 + (0.098)^2 - 2 \times 0.149 \times 0.098 = 0.0026$$

The transmission of the two coated surfaces for wave-length λ is

$$T = (0.9974)^2 = 99.5 \text{ per cent}$$

The loss is only 0.5 per cent as compared with 6 per cent in the case discussed by French.

23. Even taking into account the fact that the calculation was for monochromatic light and that the overall transmission for white light would be a little less than this, it is apparent that films of the proper thickness should be used. The experimental results of transmission experiments discussed in the next chapter further emphasize the point.

They show that those films which give rise to maximum transmission at about 5500 AU where the eye has maximum sensitivity are the most satisfactory.

Highly Reflecting Films

24. As a further example of the effect of film thickness the calculation just made can be repeated for a film of such thickness that

$$n_f t = 1/2 \lambda ;$$

i.e. waves reflected from the air-film and film-glass interfaces are in phase and their amplitudes have the same sign so that a maximum of reflected intensity by interference results. Taking values from paragraph 22 -

$$A^2 = R = (0.149)^2 + (0.098)^2 + 2 \times 0.149 \times 0.098 \cos 2\pi = 0.042$$

for wave-length λ . This value is nearly as great as the reflectivity of the uncoated glass. It is, indeed, greater than the reflectivity of a polished slab of cryolite which was found (paragraph 21) to be 0.022.

25. Awareness of these phenomena of interference led Pfund⁽⁶⁾ to a method for producing highly efficient half reflecting mirrors for use in instruments such as the Gauss eyepiece, the metallographic microscope, and the Michelson interferometer in which it is necessary that light from an external source be reflected by a mirror to an object to be viewed and subsequently transmitted by the same mirror to the eye of the observer. The ideal half reflecting mirror would reflect 50 per cent of the light and transmit 50 per cent, giving a product of reflection and transmission of 25 per cent. Glass plates, sometimes used for this purpose, reflect 10 per cent and transmit 90 per cent when the angle of incidence is 45°. The overall efficiency is thus only 9 per cent. Half silvered mirrors, while transmitting 50 per cent of the light, may reflect only 30 per cent, the remainder being absorbed, so that the product of transmission and reflection is about 15 per cent.

26. Pfund was able to produce half reflecting mirrors having a product of transmission and reflection of 23 per cent. His procedure was in every respect like that much later used in making films of low reflectivity except that a film material (zinc sulphide, $n = 2.37$) of high index of refraction and therefore of high reflecting power was chosen, and the thickness of the evaporated film was made to be such that a maximum of reflected light intensity resulted for all wave-lengths in the visible. This means only that in depositing the film the evaporation of the zinc sulphide is continued through all the color stages that appear in a vertical (wedge-shaped) soap film until eventually the film appears not colored, but white. Such a film has a higher reflecting power than a polished slab of zinc sulphide for the reasons given in paragraph 24 and absorbs very little light, so that it forms a highly efficient half reflector.

27. Pfund has given the following data:

<u>Reflector</u>	<u>Reflecting Power</u>
Single glass surface	5 per cent, calculated
Single ZnS surface	17.3 per cent, calculated
Clear cover glass (2 surfaces)	10.0 per cent, observed
Single white ZnS film on glass	31.0 per cent, observed
White ZnS film on both sides of cover glass	40.0 per cent, observed

	<u>Reflection</u>	<u>Transmission</u>	<u>Absorption</u>
Half silvered mirror	27 per cent	50	23
White ZnS on both sides of glass	41	57	2

While the efficiency of the half silvered mirror was only 13.5 per cent, that of the zinc sulphide was 23 per cent, only 2 per cent less than the theoretical maximum.

28, Pfund evaporated his films at a pressure of 5×10^{-4} mm Hg and found them to be soft and easily wiped off. Films of zinc sulphide evaporated in this Laboratory at a pressure of 10^{-5} mm were hard and resistant to rubbing. The films are not readily soluble in water and can be waxed for added protection.

29, Reverting to low reflectivity films, Cartwright and Turner^{(1),(2)} and Blodgett⁽³⁾ have given calculated curves showing reflectivity over wide spectral ranges which illustrate well the changes that take place as the wave-length is varied from values for which $n_{ft} = 1/4 \lambda$ to those for which $n_{ft} = 1/2 \lambda$ as discussed above.

Chapter III.

OPTICAL PROPERTIES: EXPERIMENTAL RESULTS

Binoculars

30. The eight interior surfaces of six sets of 7 x 50 binoculars were coated with various metallic fluoride films for the Washington Navy Optical Shop. The two external surfaces were left uncoated because at that time the vacuum system was not producing pressures sufficiently low for the production of hard films. The transmission factors of the instruments for visible light were measured at the Optical Shop, and the data were transmitted to the writer by Commander B. B. Adell. They are given in Table II.

31. The transmission factor of the uncoated binoculars was 0.55. After coating the transmission was about 0.72. The data of Table II show that generally the two barrels of each binocular were alike within the experimental error of such measurements with the exception of No. 1028 in which the transmission of the left barrel was 0.80 and of the right, 0.65. Taking 0.72 as a fair average value for the transmission factor of the coated instruments the effect of the films in reducing reflection losses can be calculated.

32. Let r be the reflection coefficient of each glass surface in the uncoated instrument. Since there are 10 such surfaces the transmission is -

$$T = \frac{I}{I_0} = (1 - r)^{10} f(a), \quad (14)$$

where I_0 is the intensity of the incident light, I the intensity of the transmitted light and $f(a)$ represents true absorption by the glass. Similarly, for the instruments in which eight surfaces were coated,

$$T' = \frac{I'}{I} = (1 - r)^2 (1 - r')^8 f(a), \quad (15)$$

where r' is the reflection coefficient of a single coated surface.

33. Dividing (15) by (14),

$$\frac{T'}{T} = \left(\frac{1 - r'}{1 - r} \right)^8 = \left(\frac{t'}{t} \right)^8 \quad (16)$$

where t and t' are the transmission by single bare and coated surfaces, respectively, and do not involve absorption by the glass. Upon putting $T' = 0.72$ and $T = 0.55$ in (16),

$$\frac{t'}{t} = 1.035.$$

If the transmission of an untreated glass surface were 0.96 ($n = 1.5$),
 that of a treated surface would be 0.993; or if t were 0.94 ($n = 1.65$),
 it would be 0.973.

4. The highest observed value of T' was 0.80 which leads to -

$$\frac{t'}{t} = 1.047 .$$

For crown glass, $t = 0.96$, $t' = 1.005$, so that the value 0.80 is higher
 than could be possible if all the coated parts were of that glass.

$$t = 0.94 \text{ (flint glass),}$$

$$t' = 0.97 ,$$

is reasonable and even a little lower than the result that might be
 obtained with high index glass because the condition of equation (12)
 (Graph 19) is more nearly met when n_g is large. The instrument
 that gave high transmission was coated with soft calcium fluoride films
 of much lower refractive index than the parent crystal; it is
 not necessarily to be concluded that transmission factors of 0.80 could
 be consistently obtained for such instruments when hard films of cryolite
 or cesium fluoride are used.

Two outer surfaces of each barrel were in all cases left
 uncoated for the reason given above that hard films could not consistently
 be placed with the equipment available when the work was done. The
 transmission that would be obtained if all the surfaces were coated can
 be calculated readily. Taking

$$\frac{t'}{t} = 1.035$$

for a single surface -

$$\frac{T'}{T} = \left(\frac{t'}{t}\right)^{10} = 1.40 = \frac{T'}{0.55}$$

$$T' = 0.77 .$$

With increasing use at sea of instruments that have been
 coated with non-reflecting films it will become clear whether outside
 surfaces should be left bare or be coated. The additional gain in trans-
 mission of 5 per cent that would have resulted by coating the external
 surfaces of these instruments is not as important as the reduction in
 reflection of stray light into the observer's eye by the eyepiece.

Transmission Measurements

In order to determine quantitatively the optical properties
 of various types of films many small squares of crown glass were coated
 on both sides and examined in stacks of five, comprising ten reflecting

* See ref.(1) for a discussion of the effect of hardness on the index
 of refraction.

surfaces. In this way the difficulties of making precise measurements on a single highly transmitting plate were avoided. In addition, a better description of the averaged effect of several films was obtained because the small inevitable differences between individual films were effective on the overall measurement as they would be in an assembled instrument. Usually, the films were deposited on the five plates of each stack simultaneously; each stack therefore represented films all nearly alike.

38. Spectral transmission curves were determined over the wavelength interval 4000 to 7000 AU with a Gaertner monochromator. A photo-voltaic cell and high sensitivity galvanometer were used to measure energies and the transmission for each set of plates was determined by taking the ratio of galvanometer deflection with the set of plates in the parallel beam of light to the deflection with the plates removed. The response of the photo-voltaic cell was linear over the intensity range that was used. The effective slit widths were about 35 AU at 4000 AU and 85 AU at 7000 AU. The measurements were sufficiently precise that the spectral transmission curves of glass filters determined with the Hardy spectrophotometer at the National Bureau of Standards could be reproduced to within ± 0.5 per cent.

39. Curves representing the transmission of several sets of bare and coated glass plates are given in Plates 4 to 9.

40. Daylight transmission measurements were made for some of the sets of plates and these are given on the right-hand side of the curves to which they apply. These measurements were made visually with a Macbeth illuminometer which contained an auxiliary blue filter to provide accurate color matching of the standard (tungsten) source and the north daylight for which the data apply. The transmission factors that were obtained represent the integrated product of spectral transmission, spectral distribution of energy in daylight, and eye sensitivity, and they are therefore a measure of the visible effect of the films on transmission.

41. For the purpose of description it is convenient to refer to the color of the films as seen in reflected light. A purple film has highest transmission for green light (λ about 5500 AU) and reflects the red and blue slightly more strongly than the green. Such a film is of the correct thickness for best performance in the visible. A thinner film shows least reflection in the blue and, appearing red in reflected light, is described as "red." In the same way, thicker films transmitting red light most copiously are described as "blue." It will be seen that the daylight transmission of the red or blue films is not greatly inferior to that of purple films, although the latter are to be preferred.

42. Plate 4 shows the transmission of five plates each coated on both sides with MgF_2 films that appeared purple in reflected light and showed maximum transmission at about 5500 AU. The transmission of the bare cover glass shows that it absorbed red light rather strongly. The origin of the glass is not known.

43. By a calculation like that of paragraph 32 the transmission of a single coated surface was obtained and is shown in the detached curve at the top of Plate 4. This curve obviously represents an average

of the individual transmissions of the 10 separate films and therefore is more representative of actual effects to be expected in instruments than either a calculated curve or a set of measurements made on one well chosen coated plate.

4. Plate 5 shows the transmission by two separate sets of MgF_2 films on crown glass, the one set purple and the other blue. The daylight transmission factors of these plates are given to the right of the figure and the reduced data for single surfaces at the top. The blue plates were quite vivid in color and transmitted λ 4000 AU very little better than the uncoated glass. The maximum value of the transmission curve is not so high (87 per cent at 6000 AU) as is the maximum to which the transmission of the purple plates of this and the preceding figure rises (90 per cent at 5500 AU), although the daylight transmission factors do not differ greatly, being 90 per cent for the purple plates and 84 per cent for the blue.

5. It was generally found that the transmission for white light measured with the eye as detector was about as great as the spectral transmission near the middle of the spectrum where the eye is most sensitive.

6. Plate 6 shows transmission curves for plates coated with CaF_2 films, the reddish purple set being too thin and the blue films, too thick. The daylight transmission factors were about the same, 83 per cent for the red and 85 per cent for the blue plates. The two transmission curves are nearly alike through the middle of the spectrum while again, seems to control the effect on vision through the coated plates.

Effect of Staggered Film Thicknesses

7. The question arises, what is the combined action of a red film on one surface with a blue film on the other surface of a glass plate? The data of Plate 6 permit a calculation, for by taking the products of the ordinates at each wave-length, the transmission by 10 plates, half of which are coated with red films and half with blue films, is obtained. The result of this operation is shown in Plate 7-a. Curve 7-b was obtained by averaging the ordinates of the transmission curve for the purple films described by Plate 5. The transmission of 10 bare glass plates is also shown.

Comparison of curves a and b shows that there is no advantage in making part of the films in a complex instrument blue and other red, for while such a procedure flattens the transmission curve and therefore leaves the transmitted light more nearly white, the use of all purple films transmitting best in the region of maximum visual acuity produces the higher transmission because the two curves are nearly alike from 400 to 4500 AU and from 6500 to 7000 AU where eye sensitivity is low.

8. If the films are to be used in a photographic instrument it may be more desirable that a combination of several thicknesses be used in order that (small) corrections to color sensitivity data of the film need not be made. The importance of the effect of a coated lens

on color values is probably not great. However, a photographic lens to be used most of the time in near infra-red, for example, might well be coated with films appropriate to that region. For λ 10,000 AU the optical thickness $n_f t = 1/4 \lambda$ would be 2500 AU. This is one-half wave-length of green light (λ 5000) and such a film would, by paragraph 24, reflect green light strongly.

50. If stacks of 5 blue and 5 purple plates are placed side by side on white paper and viewed in daylight, the difference in color of the transmitted light is easily observable. The paper appears yellowish through the blue plates and whiter through the purple plates.

51. Plate 8 shows the transmission by 5 plates coated with crystallite films, slightly too thick, and as with the best films of MgF_2 and CaF_2 described on other plates, the maximum transmission did not rise above 90 per cent which corresponds to transmission by a single surface of 99.2 per cent. This seems to be about the highest value obtainable with films on crown glass.

Water-Proofing

52. Films on MgF_2 may, after hardening (paragraph 117), be oiled or waxed, whereupon they become water-proof and, by reduction of the coefficient of friction, more resistant to abrasion. Both of these results are desirable, but they cannot be obtained without optical sacrifice. Plate 9 shows the results of a sequence of operations on one set of films. The glass plates (curve a) were coated with purple MgF_2 film on both sides and the resulting transmission curve b was of the familiar type. The plates were then waxed (Simonize) and polished with lens paper. The transmission curve c shows that while they remained superior to uncoated glass, they were decidedly inferior to the unwaxed films. After being waxed the films were pale blue in reflected light.

53. They were then reheated in a small oven to about 200°C. so that the wax evaporated. The films regained their original purple color and the new transmission curve (d) was slightly higher than the original curve. They were also as easily wet by water as before waxing.

54. The optical effect of the wax seems to be simply the addition of a layer of higher index of refraction over the non-reflecting film so that nearly as much light is lost by reflection from the wax surface as would be lost at a bare glass surface. A precise value for the index of refraction of the wax is not of importance but for such compounds of high molecular weight it varies from 1.46 to 1.48. It is possible to calculate the refractive index of the films, unwaxed and waxed, from measurements of the transmission of a stack of plates. The calculation is much simplified and not put greatly in error if the assumption is made that the light suffers no changes in intensity as the incident beam proceeds through contiguous layers of wax, film, and glass, so that at each boundary the amplitude of the reflected wave is assumed to be -

$$A = \frac{n_1 - n_2}{n_1 + n_2} ,$$

instead of more correctly -

$$A = E \frac{n_1 - n_2}{n_1 + n_2} ,$$

where E is the amplitude of the incident wave at that boundary. In other words, the amplitude of the incident wave is assumed to be unity at every interface, and since its actual departure from this value is not great, no serious error is introduced.

Index of Refraction of the Films

55. From Plate 9, the transmission factors for 5 plates of glass at 5250 AU are -

$$T_{\text{bare}} = 0.665$$

$$T_{\text{film}} = 0.885$$

$$T_{\text{waxed}} = 0.72$$

The transmission of a single coated surface is given by -

$$\frac{t_f}{t_b} = \left(\frac{T_f}{T_b} \right)^{1/10} = 1.029 .$$

The index of refraction of the bare cover glass was measured with an Abbe refractometer and found to be 1.52 so that a single surface of the bare glass reflected 4.25 per cent of the incident light. Therefore,

$$t_b = (1 - r) = 0.9575 ,$$

$$t_f = 1.029 \times 0.957 = 0.985 ,$$

and the reflection factor of the coated surface is -

$$R = 0.015 .$$

The amplitude of the resultant wave reflected from the two surfaces of the film is -

$$A = (0.015)^{1/2} = 0.123$$

if the intensity of the incident light is unity. This amplitude is the vector sum of the amplitudes reflected from the front and back (film-glass) surfaces of the film.

It is assumed that

$$A_f = \frac{n_f - 1}{n_f + 1} \quad (17)$$

$$A_{fg} = \frac{n_g - n_f}{n_g + n_f} \quad (18)$$

Since these amplitudes are in opposite phase, their resultant is given by

$$R = (A_f - A_{fg})^2$$

and this is the intensity of the reflected light.

56. Putting into (19) the expressions for A_f and A_{fg} given above and setting

$$n_g = 1.52 \text{ and } R = 0.015 ,$$

the resulting solution for the index of refraction of the magnesium fluoride films is

$$n_f = 1.4 .$$

This value seems undoubtedly to be too high - the indexes of refraction of crystalline MgF_2 for the ordinary and extraordinary rays are 1.378 and 1.390, respectively, and it is not expected that the films ever have higher refractive indexes than the massive material. The cause of the defect is obvious: since the calculation was based on the average effect of 10 separate films, none perhaps of precisely the right thickness at the wave-length chosen, the calculated value of n_f is only an apparent value for the 10 films collectively and not of great significance when applied to a single film.

57. For the sake of argument, let this value be assumed for n_f ; the apparent index of refraction of the wax required to lower the transmission from curve b to curve c of Plate 9 can be calculated under the following simplifying conditions:

The magnesium fluoride film is assumed to be covered with a layer of wax so thin that the waves reflected from the front and back surfaces of the wax are in phase, and the phase difference between waves reflected from the wax-film and the film-glass interfaces is to remain $1/2 \lambda$.

$$A_{wax} = \frac{n_w - 1}{n_w + 1} \quad (19)$$

$$A_{wax-film} = \frac{n_w - n_f}{n_w + n_f} \quad (20)$$

$$A_{film-glass} = \frac{n_g - n_f}{n_g + n_f} \quad (21)$$

Resultant reflected intensity from the composite surface is

$$R_w = (A_w + A_{wf} - A_{fg})^2 \quad (22)$$

58. From the data of Plate 9 the ratio of the reflection by a film to the reflection by a bare glass surface is given by -

$$\frac{1 - R_w}{1 - R_g} = \frac{T (5 \text{ waxed plates})}{T (5 \text{ bare plates})}^{1/10} = 1.0086 \quad (23)$$

Since

$$\begin{aligned} n_g &= 1.52, \\ R_g &= 0.0425; \end{aligned}$$

Hence, from (23),

$$R_w = 0.0343.$$

Putting this value for R_w in (22) and putting (19), (20) and (21) with numerical values for n_g and n_f in (22), the resulting value for n_w required to produce the effect of wax on the films shown on Plate 9 is -

$$n_w = 1.487.$$

This calculation is of course not exact and not of significance except that the result is in agreement with known refractive indexes of high molecular weight waxes.

59. The actual indexes of refraction of individual films are undoubtedly lower than the value 1.4 given above. The calculation of paragraph 50 has been made for a better set of magnesium fluoride films in which the average transmission of single films was 0.99. The resulting value for the index of refraction was

$$n_f = 1.35.$$

Another case of a good single plate for which the transmission of a single surface was 0.995,

$$n_f = 1.32.$$

Chapter IV.

VACUUM SYSTEM

60. The hardness of the films and the speed and ease with which they can be produced depends largely upon the excellence of the vacuum system. The principal requirements are fast pumping speed and simplicity of manual operations so that the operator can give his attention more closely to the primary function of carrying out the finishing step in the manufacture of fine optical parts.

61. The pumping system must be capable of reducing the pressure from atmospheric to 10^{-4} mm Hg within a few minutes, and this pressure must be maintained and preferably lowered during the actual evaporation of the fluorides which themselves may give up occluded gases when heated. The bell jar in which the evaporation takes place and the metal base upon which it rests present large adsorbing surfaces which give off gas during the evacuation. This condition is aggravated somewhat because such a system is comparatively dirty; each evaporation deposits fluorides on the interior surfaces, and frequent cleaning of the bell jar is required. Time so occupied will be a minimum if the pumping speed is high enough to overcome the outgassing of the walls, even when they are dirty.

62. A complete discussion of vacuum technique would be beyond the scope of this report and is not necessary. Several books on the subject are available. Of these L. Dunoyer⁽⁷⁾, Vacuum Practice (Bell, 1926) and John Strong⁽⁸⁾, Procedures in Experimental Physics (Prentice-Hall, 1938) are especially useful. The latter contains in addition to useful notes on the design and construction of vacuum systems a great deal of information concerning vacuum waxes, gaskets, and special procedures not collected so completely elsewhere. For convenience a partial list of manufacturers of special equipment is given in Appendix A.

Diffusion Pumps

63. A general description of various types of diffusion pumps has been given by Strong⁽⁸⁾. They can be made of metal or glass and contain either mercury or certain stable, low vapor pressure oils as pumping fluid. Mercury pumps require a cold trap for condensable vapors maintained at the temperature of solid CO₂ or liquid air to prevent diffusion of mercury back into the bell jar, because the vapor pressure of mercury at room temperature is about 10^{-3} mm, 10 to 100 times higher than the pressure to which the bell jar must be evacuated. While a trap cooled by solid CO₂, temperature -78°C , reduces the mercury vapor pressure sufficiently, the ice formed in the trap by condensation of moisture has at that temperature a vapor pressure of about 4×10^{-4} mm Hg and, consequently, a dish of phosphorous pentoxide must be placed in the bell jar to remove water vapor. This method is effective but slow. The difficulty can be avoided by cooling the trap with liquid air, temperature -178°C . At this temperature both water vapor and mercury are effectively removed from the high vacuum side of the system.

(8) Op. cit., Chapter 3.

64. An undesirable action of the trap is in reducing the pumping speed, for the gas must follow a devious path in traversing it rather than moving directly toward the pump. The realization of high speed requires not only that the pump be inherently fast but also that it be connected to the bell jar through tubing as short and as wide as possible with few bends and obstructions.

65. The all glass single jet mercury pump that was employed in this investigation is shown to scale in Plate 10. The drawing needs little explanation. The mercury was heated by the 220 volt nichrome heater operated on 110 volts. The stream of mercury vapor passing downward through the jet J sweeps with it air molecules that have diffused from the intake pipe into the region around the jet. The water jacket condenses the mercury vapor which then returns to the reservoir, the air being removed by a mechanical fore-pump connected to the exhaust of the diffusion pump. The trap T was cooled by filling the vacuum flask with a mixture of dry ice and alcohol, or liquid air. The arrows indicating direction of flow of the gas show how such a trap impedes it. Although more efficient traps can be constructed (several types are described by Strong*), this type was desirable in the present investigation which was conducted in part in the humid summer months when formidable collections of ice in the trap were sometimes encountered. The trap was easily warmed to free it from water by removing the vacuum flask containing the refrigerant, but this could not have been done so easily with a trap which itself served as container for the refrigerant as is the case in most of those designed for greater speed.

66. This pump was adequate for the purpose to which it was put. The lowest pressure attainable in the system was 10^{-5} mm Hg. About 15 or 20 minutes were required for the evacuation of an 18 liter bell jar to 5×10^{-5} mm.

67. Larger (faster) mercury pumps can be constructed of steel than of glass but a glass pump is more nearly free from internal disorders such as rusting, and the state of cleanliness of the pump can be determined by visual inspection.

Oil Diffusion Pumps

68. Many of the limitations of mercury pumps have been overcome by perfections in the development of oil diffusion pumps. Descriptions of several forms of these pumps have been given by Strong(8) and by Hickman(9). They offer the primary advantage of greatly increased pumping speed which comes about through the use of low vapor pressure oils as pump fluids in place of mercury so that for ordinary purposes no traps are necessary. The lowest pressures that have been obtained with such pumps without traps are, according to Hickman(9), 2.5×10^{-7} mm with multiboiler pumps containing Octoil (2-ethyl hexyl phthalate) and 5×10^{-8} mm with Octoil -S (2-ethyl hexyl sebacate). Use of a cold trap as with mercury pumps would permit lower pressures to be reached, but this is not necessary for the purpose of making evaporated films.

* Strong, p. 122.

(8) Strong, Chapter III.

69. Even though its vapor pressure is low, oil will diffuse back into the bell jar, and unless such back streaming is eliminated the oil will contaminate all parts placed in the vacuum system and bring about much trouble. Elimination of back streaming is accomplished by the use of baffles placed in the intake line and so designed as to condense oil moving toward the bell jar while decreasing the effective intake area as little as possible. Hickman⁽⁹⁾ has described such baffles and, in particular, a conical baffle discussed in more detail by Morse⁽¹⁰⁾ which they have found to be effective. The action of such baffles can be understood from the description of one type which is said to be fairly effective: it consists of a right angle, water cooled turn in the intake line of the pump.

Choice of Diffusion Pump

70. Among physicists there is no consensus at the present time as to the preferred type of pump, oil or mercury. This means that either type is satisfactory. Because of their very great pumping speeds and the success that other workers have had with them in making evaporated films, oil pumps are perhaps to be preferred. Typical performance data were recently given to the writer by Dr. R. S. Morse* who stated that at the National Research Corporation where an oil pump of capacity 100 liters/second is used, the elapsed time between placement of one set of uncoated lenses in the bell jar and the next set is seven minutes. At the commencement of the actual evaporation the pressure is 10^{-4} mm Hg and at the end it is 10^{-5} mm. This performance is excellent. Dr. Morse uses one of his own conical baffles and has had no trouble with oil entering the bell jar.

71. In recent correspondence with this Laboratory one manufacturer** described two pumps which seem to be adequate for such work. One, a glass-metal pump, was described as follows: "The glass-metal high speed diffusion pump has a speed of over 200 l/s at 10^{-4} and 10^{-5} mm, and will produce an ultimate vacuum of 10^{-5} mm, and operates efficiently at a backing pressure of 0.15 mm"

72. The second pump that they recommend is: "A recently developed all-metal vertical pump which has a speed of 300 l/s and will operate against a backing pressure of 0.10 mm and will produce an ultimate vacuum of 7×10^{-6} mm"

73. The desirable characteristics that these pumps have in common are high speed and efficient operation at high backing pressures. They will operate well with a fore pump of 0.5 liter/second capacity. Since in production work the vacuum system will be opened many times a day, it is important that the time spent in re-evacuating it to the pressure at which the diffusion pump begins to function be as small as possible. Pumps operating at backing pressures of 0.10 to 0.15 mm should be very satisfactory.

Oil Pumps

74. A mechanical oil pump is required to reduce the system to the starting pressure of the diffusion pump. The characteristics of several

telephone conversation.

Millation Products Co., Rochester, N.Y. Hickman type pump.

commercial types have been discussed by Strong⁽⁸⁾ who gives the curves shown in Plate 12. He obtained the data from the manufacturer of the pumps*. Either the Megavac (at 605 rpm) or the Hypervac 20 would be adequate for quickly reducing the pressure in the bell jar to the required fore vacuum.

75. The Central Scientific Co.** gives data from which the time required for any of their pumps to exhaust a given volume to a given pressure can be calculated. From these data the times required for evacuation of a 50-liter volume (bell jar about 35 cm diameter and 50 cm high) from atmospheric pressure to 10^{-3} mm have been computed and are given below:

<u>Pump</u>	<u>Pump Factor</u>	<u>Time to Produce 10^{-3} mm in 50 Liter Volume</u>
Hypervac 100	100	45 seconds
Hypervac 20	20	3.7 minutes
Megavac 600 rpm	6	12.5 minutes
Megavac 325 rpm	3	25 minutes
Hyvac	1	75 minutes

76. The data do not permit calculation of the time to reach 0.10 mm at which pressure the diffusion pump should begin to function, but this time would be many times less than the values given above.

77. Any mechanical pump designed for use in evacuating lamp bulbs, neon discharge tubes, etc., would serve for this purpose also if the capacity is great enough. A complete list of vacuum pump manufacturers is available in Thomas' Register of American Manufacturers. In this Laboratory both Cenco and Beach-Russ pumps have been used with success.

The Vacuum Chamber

78. The vacuum chamber and the base upon which it stands (designed by Dr. Cartwright) are shown in Plate 11, which is drawn to scale. Elaborate description seems unnecessary. The electrodes E were insulated from the brass base by glass bushings cut from Pyrex tubing and by mica washers. The electrodes were made vacuum tight with Apiezon wax W which was worked well into the clearance space between electrode, bushing, and base plate. These electrodes have given no trouble with leakage. They were designed to carry 200 amperes and are 5/8" in diameter. External electrical connections were conveniently made by plugs of 1/4" brass rod which slipped into the bored out receptacles in the bottom ends of the electrodes. These brass plugs were made by sawing a slot for perhaps 1 inch along the length of a 2 inch rod so that slight spring action was obtained. There were six electrodes in the base so that three separate filaments could be used or (as in the drawing) one electrode could serve to support the frame upon which the lenses to be coated were supported.

Central Scientific Co., Chicago.
Catalog J-136, p. 1572.

Valves and Gaskets

79. Two features of design that were introduced by Dr. Cartwright merit comment. One is the rubber gasket which serves to make a vacuum-tight seal between the bell jar and the base plate. This gasket, 1/8" square in cross-section, fits into a circular groove in the base plate 1/4" wide and 1/16" deep. When the bell jar is evacuated the pressure on the rubber becomes very high and a tight joint is assured. Removal of the bell jar after air has been admitted requires only that it be lifted off. The gasket remains in place and the procedure involves neither the labor nor the untidiness of a wax or grease joint. A suitable rubber for this purpose is Navy pure gum sheet, Navy Department Specification 55.4A*.

80. It is likely that in very large installations a large bell jar made by sealing off the end of a length of Pyrex glass tubing of suitable diameter will be desirable. Such a bell jar would not have a flanged base as does the one illustrated, and in this case it would be convenient to cut a wider groove in the base plate (or cut no groove at all) and use a wider gasket, say 1/8" thick and 1/2" wide. The narrow edges of the bell jar would produce the requisite high compression of the rubber; the edges of the gasket would tend to bow up, but it is thought that the seal would be effective.

81. Rubber gaskets can be cut by mounting the sheet rubber on a board with glue and turning this at moderate speed in a lathe, using a knife-edged tool, or a fragment of a razor blade soldered to a suitable support, lubricated with soap and water.

82. There should be on hand for emergency use a supply of Apiezon Sealing Compound Q** which, being soft and putty-like, can be pressed into place with the fingers. It can be used in place of the rubber gasket but requires more time and labor.

83. The second time saving device is the valve with which the bell jar can be sealed off from the diffusion pump, permitting the bell jar to be opened without allowing the diffusion pump to cool down. Such a valve is necessary if speed of operation is desired. The valve fits as a cap over the exhaust tube, the seal being made by a rubber gasket which fits into a groove cut around the outer edge of the cap. Three guide vanes 120° apart (two shown in the drawing) restrain the valve laterally. The supporting brass rod is allowed to move vertically by compression of the Sylphon bellows; when open, the clearance between the valve and the exhaust pipe is about 1/2". The valve is opened and held closed by the lever shown.

84. All metal joints required to be vacuum-tight were soldered. The right angle turn in the exhaust pipe was silver soldered - all other joints were made with soft solder, but traces of volatile flux, as rosin,

* This information was provided by the Manhattan Rubber Mfg. Div. of Raybestos - Manhattan, Inc. This rubber has been found satisfactory.

** This and other Apiezon waxes and greases are available from James Biddle, Philadelphia.

should not be allowed to remain inside the vacuum system, and all interior surfaces of metal parts should be cleaned of every trace of grease and dirt before assembly. Steel may be used in place of brass if it is cadmium-plated to resist rust. Iron rust is said by Strong¹ to give off 250 times as much gas as brass.

Flatness of the Base Plate

85. The base of the bell jar should seat evenly on the base plate, especially if a wax or grease seal is to be used. If the base plate is to be made of brass, a flat piece should be chosen and used without being planed or ground over the whole surface since this is likely to produce warping, but it is permissible to grind the bell jar to the plate over an annular area somewhat wider than the width of the flange on the bell jar. A steel base plate can be made flat over the entire surface by first drilling all holes and then surface grinding both sides in turn until it remains flat, afterwards cadmium plating.

86. The requirements for flatness are not critical when the rubber gasket described above is used, and materials can usually be found that require no further machining.

87. The exhaust of the system shown in Plate 11 was sealed to the intake of the diffusion pump (Plate 10) with Apiezon W wax. If the pump were metal this seal could be made with flanges on both exhaust and intake pipes clamped together with a lead fuse wire or rubber gasket between.

88. The stopcock shown in Plate 11 permitted admission of air to the bell jar and re-evacuation with a mechanical pump to the operating pressure of the diffusion pump. A bypass from the stopcock to the fore pump permitting the diffusion pump to be sealed off temporarily from both the bell jar and the fore pump while connecting the latter to the stopcock would permit use of only one mechanical pump for roughing the bell jar and maintaining the fore vacuum in the diffusion pump.

89. This stopcock, being on the low pressure side of the system, must be lubricated only with a low vapor pressure grease.

Filaments

90. The filament F which is used to vaporize the fluorides is formed by bending a platinum sheet 6 to 10 mm wide and 5 to 10 cm long into a rectangular trough left flat at the ends to permit clamping in the electrodes. Such a filament is shown in the insert on Plate 11. The thickness of the sheet may vary from 2.5 mils (0.0025 inch) to 15 mils, the thicker filaments being less susceptible to burning out by overheating when the fluoride evaporates. Platinum is chosen because it can be heated in air to fuse the fluoride powder before evacuation of the bell jar. This prevents the fluoride from blowing off the filament when it is subsequently heated in vacuum.

¹ Strong, p. 104.

Transformers

91. The filament is heated by a low voltage transformer capable of supplying 200 amperes at about 2 to 5 volts. The currents required to heat three different filaments to the temperature required for evaporating Hg are given below:

<u>Size</u>	<u>Current</u>
8 mm x 40 mm x 2.5 mil	45 amperes
7 mm x 55 mm x 15 mil	120 amperes
11.5 mm x 40 mm x 15 mil	More than 150 amperes

It is convenient if the transformer has taps for about 5, 10, and 20 volts since other filaments, as those of tungsten used in evaporating aluminum, require higher voltages than platinum.

92. The current was controlled by a Variac* transformer in the input line of the filament transformer.

Vacuum Gauges

93. The pressure should be measured with a continuously reading vacuum gauge. For details of construction and operating characteristics of the several gauges available the reader is referred to Strong⁽⁸⁾ or Dunoyer⁽⁷⁾. Three types are useful. The thermocouple gauge**, consisting of a hot wire near a thermoelectric junction so that the temperature of the wire and the thermocouple response become greater as the pressure is lowered, is useful over the pressure range 10^{-2} to 10^{-3} or 5×10^{-4} mm Hg. At 10^{-3} mm it is safe to turn on an ionization gauge which functions to 10^{-8} mm. These two gauges used together form a complete system. While the thermocouple gauge can be dispensed with in a vacuum system functioning properly because the operator soon learns from experience, by watching the glow discharge excited by a high frequency induction coil, by the sound of the mechanical pump, in some oil pumps by the place at which condensation of oil occurs, to estimate the higher pressures. The thermocouple gauge is useful in hunting leaks and assures that the ionization gauge will not be burned out by being turned on at too high a pressure.

94. The ionization gauge is a triode vacuum tube (radio tubes can be adapted to the purpose, but not with complete reliability) consisting of the usual filament, grid, and plate. An applied potential between filament and grid produces an electron current which on entering the space between grid and plate ionizes the gas there by impact. When a small potential is applied, the ionization current between plate and grid is measured with a microammeter or galvanometer and decreases linearly with the pressure below about 10^{-4} mm. A well designed ionization gauge*** has been described recently by Morse and Bowie⁽¹¹⁾. Type FP-62 vacuum tubes also are designed for this use. Circuits for operation of the gauges are available in the literature or obtainable from the manufacturers of the tubes.

* Type 100-K, load 2 KVA (General Radio Co.).

** General Electric Co.; Central Scientific Co.

*** Available from Distillation Products Co.

95. The Pirani gauge consists in its simplest form of two lamp bulbs, one highly evacuated and sealed off and the other connected to the vacuum system, in the arms of a Wheatstone bridge circuit. The filaments are heated by the bridge current and that one exposed to air in the vacuum system becomes hotter and of higher resistance as the pressure is reduced, so that the unbalance of the bridge registered by a microammeter is a measure of the pressure. The useful range of such a gauge is 0.1 to about 5×10^{-5} mm, but reliable measurements near the lower limit of the scale require careful calibration against a McLeod gauge. Although the ionization gauge is much to be preferred for this work, a Pirani gauge may suffice for production work. Since evaporation will usually be commenced at 10^{-4} mm a gauge reading reliably to about 5×10^{-5} mm would perhaps be satisfactory. However, neither the thermocouple gauge nor the Pirani gauge that were used in part of this investigation were considered to be reliable enough toward the lower ends of their ranges. Pressures were ultimately read with a McLeod gauge which is not recommended for production work because it is not continuously reading.

CONCLUSION

96. Any vacuum system, no matter how well designed, will require repair and expert attention from time to time. Two courses may be followed in successfully establishing production units:

- (a) Complete units may be purchased from institutions experienced in the design and construction of such equipment* and operated by personnel previously experienced in such work or capable of acquiring the necessary experience, or preferably
- (b) Experienced vacuum technicians may be engaged who, given the necessary pumps, can design their own vacuum systems, each to his own liking.

97. Improvement in production practice and more nearly continuous operation of the production units will result if small auxiliary evaporation apparatus is available for experimentation.

98. The reader of this incomplete discussion of vacuum technique will appreciate a remark by Hickman**": "Clearly, the means of producing an empty cabinet neatly and economically still eludes the engineering physicist."

Distillation Products Co., Rochester, N.Y.; Central Scientific Co., Chicago; Baird Associates, Cambridge, Mass.; National Research Corporation, Boston; Vard-Mechanical Laboratories, Pasadena; C. H. Cartwright, 46 West Fourth St., Corning, N.Y.; Evaporated Films Co., Ithaca, N.Y.; and perhaps others of whom the writer has no knowledge.

Loc. cit., p. 311.

Chapter V.

THE EVAPORATION PROCESS

Cleaning the Glass

99. The glass parts to be coated must be chemically clean. Rubber gloves should be worn to prevent contamination of the clean surface with oil from the fingers. The glass is first washed with a clean cotton swab and soap and water or some detergent such as Dreft or Alphasol. After rinsing with water the glass is washed in sodium or potassium hydroxide solution, and then in nitric acid. The glass will not be damaged by the cleaning solutions if they are applied only for the time required for cleaning. Glass left in the solutions may be etched. A safe and effective method of applying the cleaning solutions is to rub the surface of the glass, already wet with water, briefly but thoroughly with a finger (in rubber glove) that has been dipped in the alkali or acid bath. The lens is then flushed immediately with water and wiped dry. If the water is allowed to evaporate streaks will remain on the glass even when distilled water is used.

100. Ordinary paper towels have been found satisfactory for blotting off the excess water. No scratching of the glass has been encountered because care has been taken not to rub very hard with the paper. Final traces of water can be removed with lens paper. Lint left by the paper is removed by a method described below.

101. In place of paper the lenses may be wiped dry with cloth, but it must have been laundered a great many times to remove natural oils. Old linen is perhaps to be preferred to cotton. Cloth does not leave lint, and its use eliminates the troublesome operation of removing it; cloth, however, is more likely to recontaminate the surface than paper because the latter is discarded after being used while the cloth, if used many times, will eventually become dirty again. Since "dirtiness" as such here is not something that can be seen, the first knowledge that the cloth is dirty is given by the appearance of spots and streaks on the evaporated film, often some time after it has been removed from the bell jar. Small squares of cloth, each piece being used only one time (or more often, as experience may prove), and dispensed from a suitable receptacle, would be satisfactory.

Removal of Lint and Dust

102. Special camel's hair brushes constructed by cementing the bristles around the outer edge of a brass tube (inside diameter 1/16 to 1/8 inch) with Insoluble cement are used by the Washington Navy Yard Optical Shop. The hollow handle of the brush can be connected to a small vacuum pump such as a water aspirator or mechanical suction pump by rubber tubing, and when it is used in this way lint and dust can be removed with the gentlest touch. The brush can be washed with organic solvents to remove grease, and it is the most effective device the writer has seen for removing dust. If very carefully washed and subsequently protected from any source of dirt it should be useful in the final step of cleaning the lens.

103. Hair brushes, however, cannot be subjected to the complete cleaning that can be given to glass. Such a brush* has been used by the writer sometimes with success and at other times with the discouraging result of leaving oil films on the glass. The glass appeared to be clean, but spots sometimes resulted after the films had been deposited. A substitute for the vacuum brush is a brushless nozzle for the vacuum cleaner made by drawing down the end of a glass tube to leave a small opening about one-half or one millimeter in diameter. Such a tip for the vacuum cleaner can be washed in acid to make it chemically clean. While it is not quite as effective as the brush, only the most tenacious dust particles resist it, and these can be dislodged by brushing lightly with the glass tip. The rounded fire-polished end will not injure the surface of the lens.

104. After the glass has been cleaned it should be placed in the bell jar and coated with its non-reflecting film as promptly as possible because it will soon become covered with a monomolecular layer of oil if left exposed to the air.

Testing for Cleanliness

105. The standard and perhaps the best test for cleanliness of a glass surface is to observe the uniformity of the vapor deposit formed when the glass is breathed upon. Non-uniform breath figures show where the dirt lies, for water condenses to form a transparent invisible film on clean glass, but a gray visible film on dirty surfaces. However, the breath itself may contaminate the surface, and it is better after careful chemical cleaning to observe whether water spreads uniformly over the glass. A drop of water should wet chemically clean glass well enough to flow over the entire surface. If the film of water draws away from the edges or from localized areas of the surface the cleaning process must be repeated. The film of water will evaporate uniformly over the entire surface of clean glass, and when it becomes sufficiently thin, interference colors like those produced by thin films of oil can be seen. If interference colors can be seen after the water has evaporated they are due to oil on the glass, which must be recleaned.

Cleaning with Electric Discharge

106. Glass cleaned by the method that has been described is usually clean enough. After it has been placed in the vacuum chamber further cleaning can be effected by producing an electric discharge in the gas while the system is being pumped out. This is done conveniently with a high frequency induction coil (leak detector) mounted outside and in contact with the glass bell jar. Such an induction coil is a necessary adjunct to vacuum equipment because of its usefulness in finding the source of trouble when leaks develop or the pumps do not function properly. With the system operating properly it serves the double purpose of giving the operator a visual means of following the progress of the evacuation and of producing final cleaning of the glass surfaces by ionic bombardment. That this cleaning method is effective for traces of dirt is best shown by the common experience that glass on which metals are to be sputtered with a high

* Provided by Mr. A. J. Devlin, Optical Shop, Washington Navy Yard.

tial discharge does not require to be so carefully cleaned as glass which films are to be evaporated.

Preliminary Treatment of Glass

107. Glass that has been freshly polished is easily cleaned, but it is wiped with organic cleaning agents such as xylol or benzene which do not spread a thin layer of oil over the surface, or if a layer of oil has been left on the surfaces of cemented lenses, the chemical cleaning process becomes difficult to carry out. Cemented lenses should be freed of every trace of excess balsam by wiping with absorbent cotton and alcohol. By far the most difficult contaminant to remove has proved in this investigation to be Canada balsam. In general, the glass surface should be left as nearly untouched after it leaves the lap as is possible.

108. Cemented lenses and prisms that have had one face silvered and painted present another difficulty: Canada balsam or paint, unless well dried, may have vapor pressures so great that evacuation of the system to the required low pressure of 10^{-4} to 10^{-5} mm is impossible. There are, in fact, very few organic materials that can be used inside a vacuum apparatus and these are the special waxes, greases and oils used just for that purpose. A paint which can be used to cover the surfaces of cemented lenses and which can be put on over the special protective lacquer used on silvered surfaces is Glyptal* lacquer. This lacquer when used at room temperature has a vapor pressure only 8.5 times that of water (12). It may turn out not to be necessary to paint over the balsam with protective black paint when very large pumps are used and reasonable evaporation of these volatile compounds is allowed. In the course of this investigation both old and new optical parts presenting free surfaces were coated with balsam and paint were coated. There was never any difficulty in raising the pressure with old parts or new parts lacquered with Glyptal; there was always difficulty with unlacquered new parts, both with Canada balsam and protective black paint.

Position of the Films

1. The platinum filament, Plate 11, is filled with the fluoride powder and heated in air until all the powder is fused. Unless this is done the powder will blow off the filament when it is heated in vacuum**. The parts to be coated are then put into position on their supporting blocks (Plate 11) and, unless the lenses and their support fill nearly the whole cross section of the bell jar leaving few interstices through which fluoride vapor can pass, they are covered over with a watch glass to prevent irregular deposition of the film material on their top surfaces.

General Electric Company.

When it is prefused MgF_2 sometimes explodes when the evaporation is begun, depositing small specks of the material on the lens. These are hard to remove, but they can be prevented by heating the fluoride slowly or by arranging a shield over the filament to be removed by manual control or by means of a ground joint in the base when the fluoride is hot and the fluoride has begun to evaporate.

109. If several lenses are to be coated at one time the support should be bent into a rough spherical segment so that each lens faces squarely toward the filament. A distance of 25 or 30 cm above the filament is suitable for producing fairly uniform films on lenses distributed over a 10 cm diameter support.

110. While the bell jar is being evacuated the filament is outgassed by being heated to redness (but not to the melting point of the filament), so that water vapor and trapped gases are driven off. This may continue through almost the entire period of evacuation if the pump is fast. Not more than two or three minutes are required for freeing the filament of gas, and if it is not to be left warm through the entire evacuation it were better heated after the pressure has been reduced considerably than at the beginning of the pumping.

111. When the pressure reaches 10^{-4} mm the filament is heated until zinc fluoride melts and within 30 seconds or a minute as the operator chooses, a film will be made. Practice soon establishes the necessary experience required for controlling filament temperature. The evaporation need only to proceed slowly enough that the growth of the film can be watched easily. When the correct film thickness is nearly reached the temperature may be slightly reduced to permit more accurate termination of the evaporation.

Measuring the Film Thickness

112. The growth of the film is watched by observing the color changes in white light reflected from the surface. A frosted electric bulb is partly painted over the surface so that a rectangular area about 10 cm is left bare is a convenient source. The clearly defined edges of the rectangle enable the observer to distinguish between images reflected from the two surfaces of plane parallel parts or from the two reflecting faces of a totally reflecting prism. When lenses are being coated, the images from top and bottom surfaces can always be identified and an unpainted light bulb is adequate.

113. The sequence of colors through which the reflected light passes as the film increases in thickness is that seen in a vertical (wedge shaped) interference film. A very thin film appears gray. As it grows thicker the light is yellowish and then red when the thickness producing least reflection of light is reached. When the optical thickness becomes equal to one-half wave-length of green light near the middle of the spectrum the reflected light is purple, and at this point the evaporation should be stopped. The purple color should be slightly more blue than red, but the judgment for precision is not great; if the film appears to the observer as purple, not distinctly red or blue, it will be satisfactory.

114. If the thickness is allowed to grow further the film will appear red, signifying maximum transmission for red light. If the evaporation is continued beyond this point the color sequence begins over again. When the thickness becomes equal to three-quarter wave-lengths of blue light, the film will eventually reach a thickness for which the phase difference between wave-lengths at the ends of the spectrum is not great, and the film will appear white. This is the required condition for a reflecting film of zinc sulphide (paragraph 24 ff.).

Physiological Note

115. All the metallic fluorides are protoplasmic poisons and care should be exercised to prevent their entering the body, especially by breathing when the powder is being fused on the filament while the system is open to the air. The bell jar or another glass vessel can be placed over the filament to trap the material that comes off the filament during this process. When the bell jar is removed after the evaporation a strong odor can sometimes be detected. It is not known to what extent the materials decompose in the course of the evaporation, but whatever the form of the vapor, fluoride or fluorine, its effect is to produce unpleasant bronchial irritation which can be the prelude to infection. The question of placing ventilating hoods over production units should not be passed over without consideration by those competent to say how great the danger of prolonged exposure may be. A simple respirator with a filter for trapping dust and condensable vapors has been found to remove most of the odor from the hot salt and, presumably, most of the danger.

Chapter VI.

MECHANICAL PROPERTIES OF THE FILMS

Hardness

116. The evaporation must be made at pressures less than 10^{-4} mm if hard films of MgF_2 and cryolite are to be produced. Films of CaF_2 seem always to be soft. The others, MgF_2 and cryolite, are hard enough to withstand handling and gentle rubbing with lens paper or soft cloth. They will not bear abrasion or vigorous rubbing since even the parent crystals are relatively soft (Table III). This table shows that MgF_2 , hardness 5, forms more durable films than cryolite, hardness 2.5.

Hardening by Heat

117. Magnesium fluoride films, although hard enough for purposes of assembly, can be made even harder by heating to about $400^\circ C$. after which they can hardly be removed by rubbing with cloth, provided the glass upon which they are deposited is clean. Heating of good optical parts should be carried on under such conditions that strains will not be set up. For most purposes it is not necessary that the films be so hardened unless they are to be on surfaces exposed to dirt and subject to frequent cleaning. The only measure of durability of such exposed films of which the writer has knowledge is that on ordinary eye glasses, subject to frequent cleaning and constant exposure to the elements, they remain for about 8 months before thin spots begin to appear.

Hardening with Age

118. Films of MgF_2 generally become harder with age, but not so hard as when they are heated. In addition, the surfaces probably acquire monomolecular films of oil from their surroundings, and this improves their durability without producing the decrease in light transmission discussed in paragraph 52 ff.

Effect of Water

119. MgF_2 films have been soaked in water for several days with no impairment of their optical properties but in every case with softening of the film. The films so softened regain their hardness upon reheating, but it is not definitely known if they become hard again with age. The problem is not serious for optical parts mounted inside an instrument provided they do not have to be taken out for frequent cleaning. Condensation of moisture in normal use should not seriously affect them. It remains to be seen how these films withstand the various phases of use under service conditions.

120. It has been observed, however, that films freshly deposited are subject to rapid and serious attack by moisture. MgF_2 films deposited on wet days when the relative humidity was 80 per cent often frilled and became unusable immediately upon opening the bell jar. Films made on dry days when the relative humidity was 50 or 60 per cent have never been observed to deteriorate upon subsequent exposure to

moisture. Also, films made on a very wet day but allowed to remain in the evacuated bell jar over night were not damaged when exposed to air on the following very humid day. The available experience does not permit a decision as to whether an air conditioned room should be specified as a necessary environment for successful manufacture of the films on humid days because the effects that have been described were observed in only a few cases on the wettest days. CaF_2 films evaporated on wet days were stable.

Chapter VII.

SUMMARY AND CONCLUSIONS

121. The detail in which this report has been written is perhaps justified not by any novelty of the fundamental phenomena upon which the action of the films is based nor because knowledge of vacuum technique is so widespread but rather because many of the subjects that have been discussed are those about which questions were continually arising. The discussion of many of these things is incomplete; it is expected that many simplifications and improvements in technique will evolve in establishments where production units are put into operation and that much additional information about the optical and mechanical properties of the films will be acquired. The summary that follows, accordingly, emphasizes those things that are thought to be fundamental.

Diffusion Pumps

122. It is recommended that oil diffusion pumps be used rather than mercury pumps for these reasons:

- (a) Pumping speeds of 200-300 liters/second are easily obtained so that minute leaks and adsorption of gases by the walls of the system are not so likely to be sources of trouble.
- (b) Since these pumps require no traps, they produce a dry vacuum, the water being pumped out along with other gases. Although a mercury pump with a trap cooled with liquid air would also remove water vapor, the use of dry ice would not. The oil pump eliminates the necessity for obtaining liquid air from outside sources.

123. Oil pumps may be purchased or they can be built by personnel trained to superintend the work. It must not be concluded that the performance of any pump or arrangement of apparatus described in this report cannot be satisfactorily met by other equipment differing greatly in design. Most persons who have engaged in vacuum work have at one time or another built pumps for special purposes, and there are many other designs.

Mechanical Pumps

124. As examples, the Central Scientific Company Megavac 600 rps, capacity 0.5 liter/second at 10^{-2} mm, or the Hypervac 20, capacity 1.5 liters/second at 10^{-2} mm are adequate for producing the necessary forevacuum.

Valves and Gaskets

125. The valve shown in Plate 11 for closing off the diffusion pump from the bell jar so that consecutive evaporations can be made without waiting for the pump to cool has been very successful and is considered a necessary part of any equipment designed for fast work.

126. The narrow rubber gasket (Navy pure gum sheet, Specification 120) used for sealing the bell jar to the base plate has been

satisfactory and has given less trouble with leakage than any other (type) rubber gaskets or wax seals.

Pressure Gauges

127. It is recommended that ionization gauges be used to measure the ultimate low vacuum required for the evaporation of hard films with an inexpensive thermocouple gauge for measuring pressures above 10^{-5} mm where it is not safe to operate the ionization gauge.

Film Materials

128. Since magnesium fluoride forms the hardest films, it is recommended that this material be used (with comparative experiments on corundum highly desirable). Hardening of the films by heat is not recommended since if they are deposited at low pressures they are hard enough to permit assembly of instruments without damage. It is not recommended that the films be waxed to make them waterproof unless subsequent experience with instruments at sea shows that this opinion should be altered. Exposed surfaces may be either coated with the films or left bare. If coated, the films probably will be removed by cleaning unless they are hardened by heat, but this would not interfere with use of the instrument.

New Research

129. Since laboratory experience with problems of this sort cannot pre-determine completely the difficulties of production, it is recommended that continued experimentation be carried on in the places where production work is being done, hence, where new problems arise. The literature on the subject of reducing the reflection of glass by the use of evaporated films is meager, but new information can be expected to appear with increasing frequency. It is desirable that new suggestions be investigated under conditions that permit comparison with existing practices, and this can best be done in the laboratories of the optical shops engaged in the work, and where, in fact, many of the improved practices should originate.

Table I

Transmission by Glass Plates.
(No absorption losses are included.)

Number of Surfaces	Refractive Index			Crown Glass Coated with MgF_2
	1.50	1.575	1.65	
1	0.96	0.95	0.94	0.99
2	0.92	0.90	0.88	0.98
4	0.85	0.81	0.78	0.96
6	0.78	0.73	0.67	0.94
8	0.72	0.66	0.61	0.92
10	0.66	0.60	0.54	0.90
12	0.61	0.53	0.45	0.88
14	0.56	0.48	0.42	0.87
16	0.52	0.43	0.37	0.85
18	0.48	0.39	0.33	0.84
20	0.44	0.36	0.29	0.82
22	0.40	0.33	0.24	0.80
24	0.37	0.28	0.20	0.78

Table II

Transmission Factors of 7x50 Binoculars
Coated with Metallic Fluoride Films.*

<u>Number</u>	<u>Film Material</u>	<u>Left Barrel</u>	<u>Right Barrel</u>
1010	Magnesium fluoride	0.71	0.72
1023	Magnesium fluoride	0.73	0.72
1009	Calcium fluoride	0.74	0.72
1028	Calcium fluoride	0.72	0.80
972	Cryolite	0.75	0.71
1006	Cryolite	0.72	0.71

Transmission by uncoated binoculars - 0.55

* These data were provided by the Optical Shop, Washington Navy Yard.

Table III

Properties of Metallic Fluorides.

<u>Name</u>	<u>Formula</u>	<u>n_o, n_e</u>	<u>Melting Point, °C.</u>	<u>Boiling Point</u>	<u>Solubility in grams per 100 cc cold water</u>	<u>Hard- ness</u>
Magnesium fluoride	MgF ₂	1.378, 1.39	1396	2239	0.0076	5
Calcium fluoride	CaF ₂	1.434	1360	-	0.0016	4
Sodium aluminum fluoride	3NaF·AlF ₃	1.364	1000	-	sl. s.	2.5

These data are from the Handbook of Chemistry and Physics (Chemical Rubber Co.), 23rd ed., p. 360 ff.

The hardness scale is:

1 Talc	6 Feldspar
2 Rock salt	7 Quartz
3 Calcite	8 Topaz
4 Fluorite	9 Corundum
5 Apatite	10 Diamond

satisfactory and has given less trouble with leakage than any other (e) rubber gaskets or wax seals.

Pressure Gauges

127. It is recommended that ionization gauges be used to measure ultimate low vacuum required for the evaporation of hard films with an inexpensive thermocouple gauge for measuring pressures above 10^{-5} mm. It is not safe to operate the ionization gauge.

Materials

128. Since magnesium fluoride forms the hardest films, it is recommended that this material be used (with comparative experiments on quite highly desirable). Hardening of the films by heat is not recommended since if they are deposited at low pressures they are not enough to permit assembly of instruments without damage. It is recommended that the films be waxed to make them waterproof unless frequent experience with instruments at sea shows that this opinion should be altered. Exposed surfaces may be either coated with the wax or left bare. If coated, the films probably will be removed by cleaning unless they are hardened by heat, but this would not interfere with use of the instrument.

Research

129. Since laboratory experience with problems of this sort cannot pre-determine completely the difficulties of production, it is recommended that continued experimentation be carried on in the places where production work is being done, hence, where new problems arise. The literature on the subject of reducing the reflection of glass by the use of evaporated films is meager, but new information can be expected to appear with increasing frequency. It is desirable that new suggestions be investigated under conditions that permit comparison with existing practices, and this can best be done in the laboratories of the optical shops engaged in the work, and where, in fact, many of the improved practices should originate.

Table I

Transmission by Glass Plates.
(No absorption losses are included.)

Number of Surfaces	Refractive Index			Crown Glass Coated with MgF_2
	1.50	1.575	1.65	
1	0.96	0.95	0.94	0.99
2	0.92	0.90	0.88	0.98
4	0.85	0.81	0.78	0.96
6	0.78	0.73	0.67	0.94
8	0.72	0.66	0.61	0.92
10	0.66	0.60	0.54	0.90
12	0.61	0.53	0.45	0.88
14	0.56	0.48	0.42	0.87
16	0.52	0.43	0.37	0.85
18	0.48	0.39	0.33	0.84
20	0.44	0.36	0.29	0.82
22	0.40	0.33	0.24	0.80
24	0.37	0.28	0.20	0.78

Table II

Transmission Factors of 7x50 Binoculars
Coated with Metallic Fluoride Films.*

<u>Number</u>	<u>Film Material</u>	<u>Left Barrel</u>	<u>Right Barrel</u>
1010	Magnesium fluoride	0.71	0.72
1023	Magnesium fluoride	0.73	0.72
1009	Calcium fluoride	0.74	0.72
1028	Calcium fluoride	0.72	0.80
972	Cryolite	0.75	0.71
1006	Cryolite	0.72	0.71

Transmission by uncoated binoculars - 0.55

* These data were provided by the Optical Shop, Washington Navy Yard.

FEB 27 1941

Table III

Properties of Metallic Fluorides.

<u>Name</u>	<u>Formula</u>	<u>n_o, n_e</u>	<u>Melting Point, °C.</u>	<u>Boiling Point</u>	<u>Solubility in grams per 100 cc cold water</u>	<u>Hard- ness</u>
Magnesium fluoride	MgF ₂	1.378, 1.39	1396	2239	0.0076	5
Calcium fluoride	CaF ₂	1.434	1360	-	0.0016	4
Sodium aluminum fluoride	3NaF·AlF ₃	1.364	1000	-	sl. s.	2.5

These data are from the Handbook of Chemistry and Physics (Chemical Rubber Co.), 23rd ed., p. 360 ff.

The hardness scale is:

1 Talc	6 Feldspar
2 Rock salt	7 Quartz
3 Calcite	8 Topaz
4 Fluorite	9 Corundum
5 Apatite	10 Diamond

APPENDIX A

SOURCES OF SUPPLY

A. Metallic Fluoride Powder

Eimer and Amend, New York, N.Y.
Baker & Co., Phillipsburg, N.J.

B. Platinum and other metals for filaments

Baker & Co.
American Platinum Works, Newark, N.J.

C. Oil Diffusion Pumps

Distillation Products Co., Rochester, N.Y.

D. Mechanical Pumps

Central Scientific Co., Chicago, Ill.
Kinney Mfg. Co., Boston, Mass.
Beach-Russ, New York, N.Y.
And others listed in Thomas' Register of American Manufacturers.

E. Vacuum Waxes, Oils and Greases

Octoil
Celvacene grease } Distillation Products Co.

Apiezon "W" wax
"Q" sealing compound } James Biddle, Philadelphia.
Apiezon stopcock grease
Picein wax

Glyptal lacquer - General Electric Co.

Cello seal stopcock grease - Fischer & Co., Pittsburgh.

F. Bell Jars

Central Scientific Co.
Eimer and Amend
Chicago Apparatus Co.
and others.

For special large bell jars consult Corning Glass Co., Corning, N.Y.

G. Pressure Gauges

Ionization gauges }
Pirani gauge } Distillation Products Co.

(Continued)

G. (Continued)

Thermocouple gauge { Central Scientific Co.;
General Electric Co.,
Special Products Division.

H. Sylphon Bellows

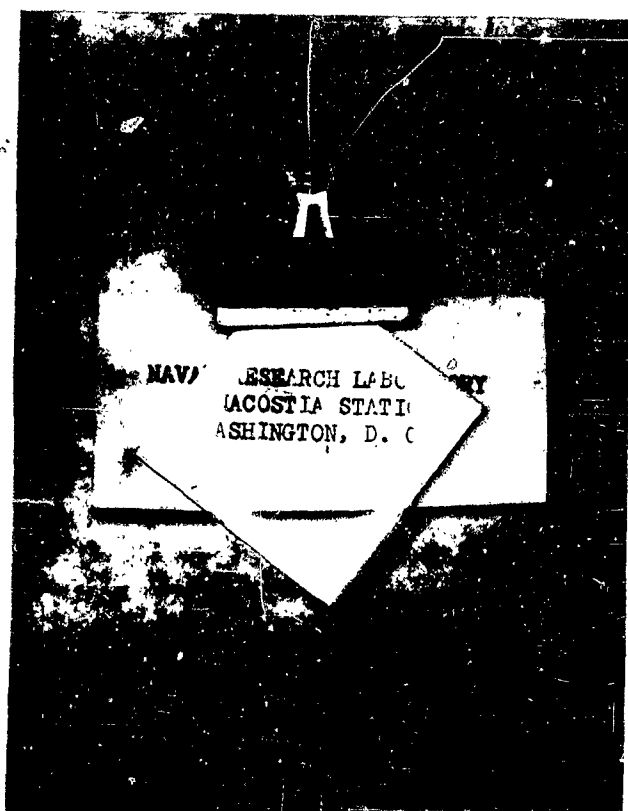
Fulton Sylphon Co., Knoxville, Tenn.

I. Hydron Bellows

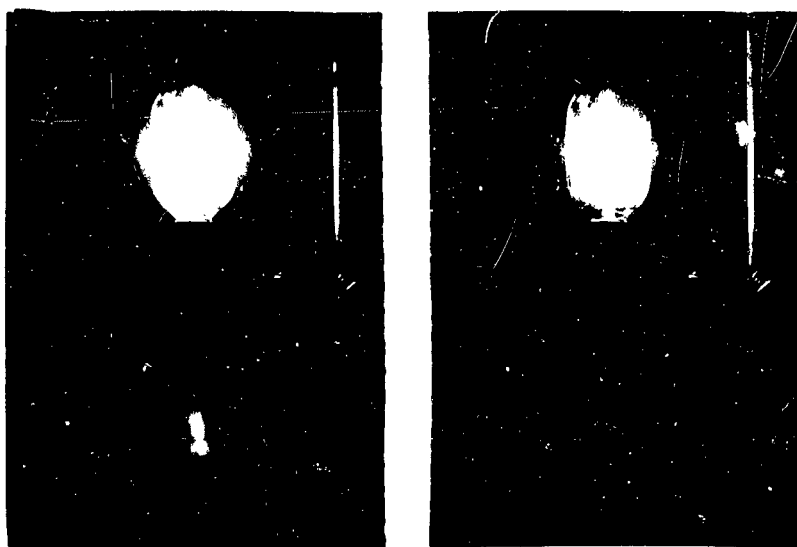
Clifford Mfg. Co., Boston, Mass.

REFERENCES

1. C. H. Cartwright and A. F. Turner, "Process of Decreasing Reflection from Surfaces, and Articles so Produced," U.S. Patent No. 2,207,656 (July 9, 1940).
2. A. F. Turner, "Coating Lenses," Phototechnique, October 1940, p. 48.
3. Katherine B. Blodgett, "The Use of Interference to Extinguish the Reflection of Light from Glass," Phys. Rev. 55, 391 (1939).
4. F. L. Jones and H. J. Homer, "Chemical Methods for Increasing the Transparency of Glass Surfaces," Jour. Opt. Soc. Am. 30, 654 (1940) A; the complete paper appeared in the same journal 31, 34 (1941).
5. J. W. French, "Cryolite Films on Glass Surfaces," Nature 146, 687 (November 23, 1940).
6. A. H. Pfund, "Highly Reflecting Films of Zinc Sulphide," Jour. Opt. Soc. Am. 24, 99 (1934).
7. L. Dunoyer, Vacuum Practice, Bell, 1926.
8. John Strong, Procedures in Experimental Physics, Prentice-Hall, 1938.
9. K.C.D. Hickman, "Trends in Fractionating Pumps," Physics 11, 303 (1940).
10. R.S. Morse, "Baffles for Oil Diffusion Pumps," Rev.Sci.Inst. 11, 277 (1940).
11. R. S. Morse and R. H. Bowie, "A New Style Ionization Gauge," Rev. Sci. Inst. 11, 91 (1940).
12. T. P. Sager and R. G. Kennedy, Jr., "The Use of Glycol-Pthalic Anhydride Resin as a High-Vacuum Cement," Physics 1, 352 (1931).



Photograph through two glass plates
coated with Mg F₂ over the central area.

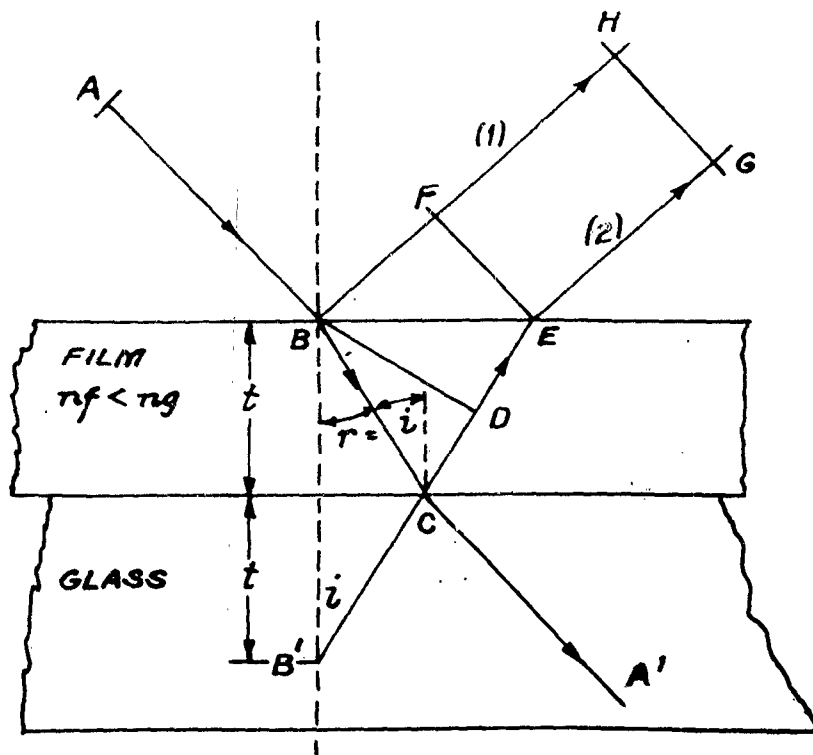


Photographs of 500 watt light with
zeiss f:4 planar lens, 8 surfaces.
Left: Uncoated. Right: Coated with C₂F₂.

Plate 2

27 '941

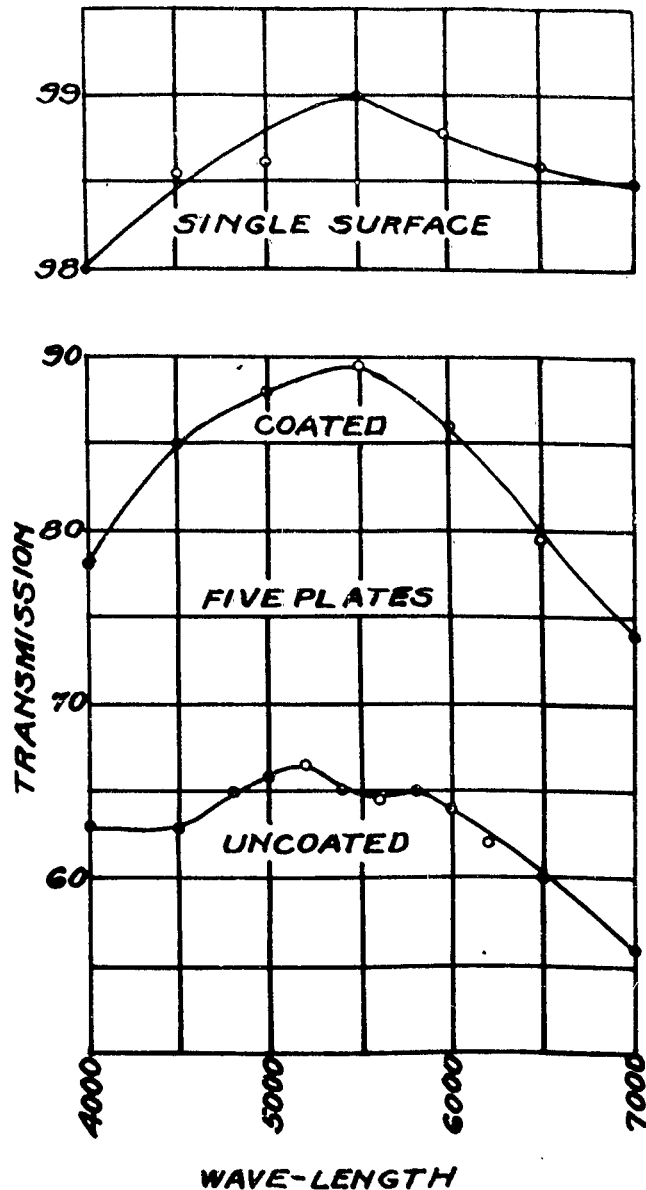
PHASE RELATION BETWEEN WAVES REFLECTED FROM
THE FRONT AND BACK SURFACES OF A THIN FILM.



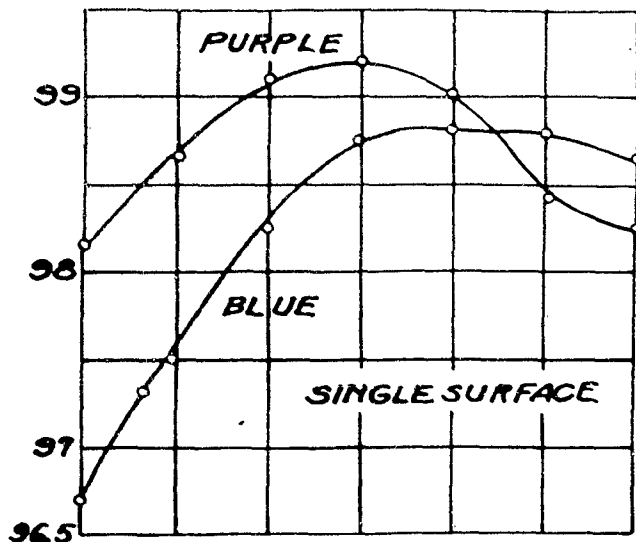
OPTICAL PATH OF (1) = $AB + BF + FH$;
OPTICAL PATH OF (2) = $AB + n_f (BC + CE) + EG$.
 $(2) - (1) = n_f (BC + CE) - BF$.

DRAW BD PERPENDICULAR TO CE ; THEN $BF = n_f (DE)$.
DROP A PERPENDICULAR FROM B AND EXTEND EC
TO B' SO THAT $BB' = 2t$ AND $B'C = BC$.

THEN $(2) - (1) = n_f [BC + (CE - DE)] = n_f (B'C + CD) = n_f (B'D)$
 $= 2 n_f t \cos i$.



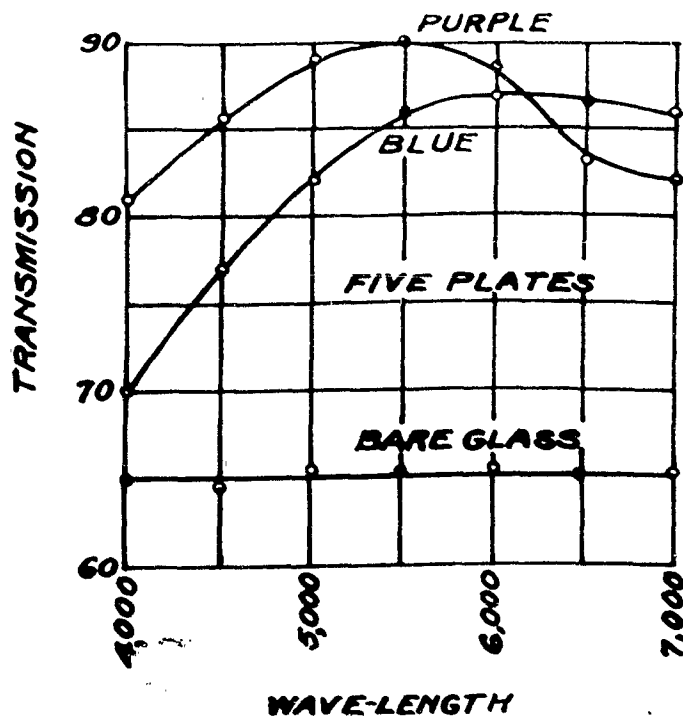
TRANSMISSION BY MgF_2 FILMS



T (DAYLIGHT)

PURPLE 99.1

BLUE 98.5

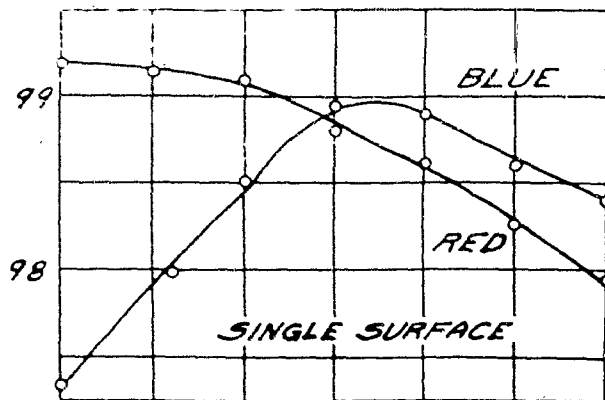


T (DAYLIGHT)

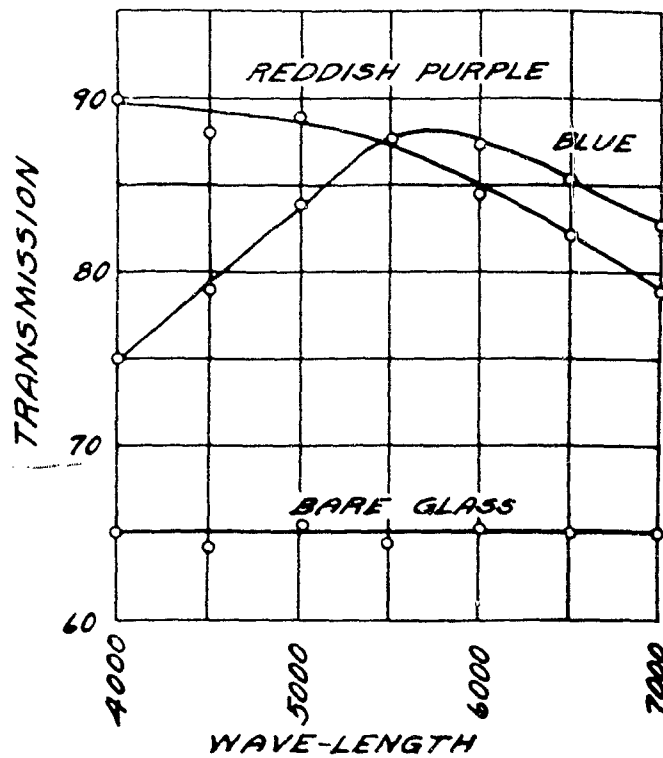
PURPLE 0.90

BLUE 0.84

TRANSMISSION BY MgF_2 FILMS.

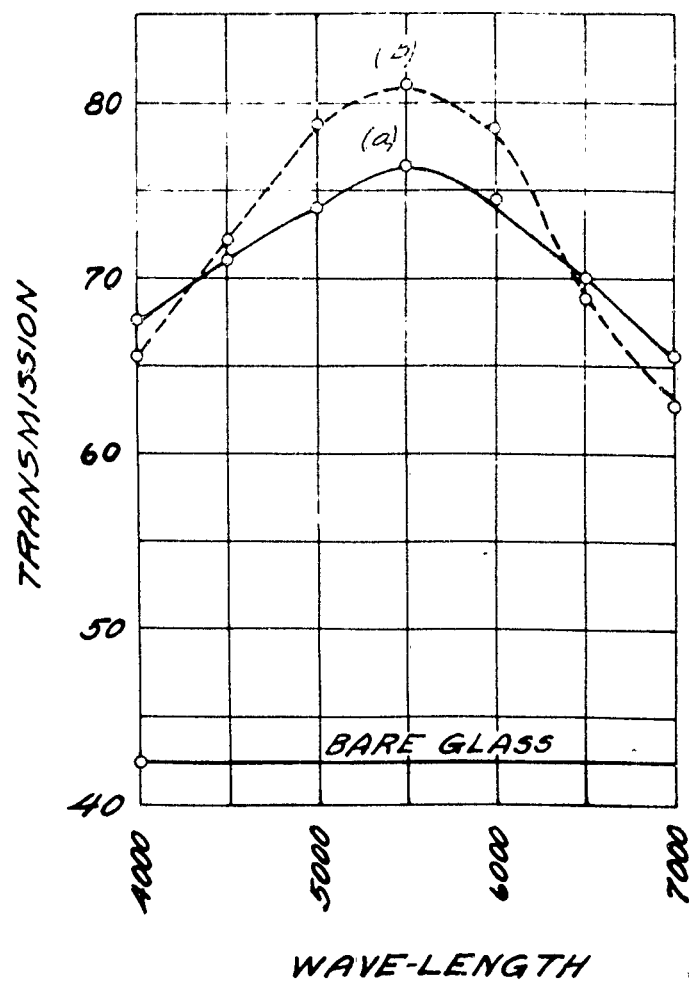


T (DAYLIGHT)
RED 98.4
BLUE 98.6

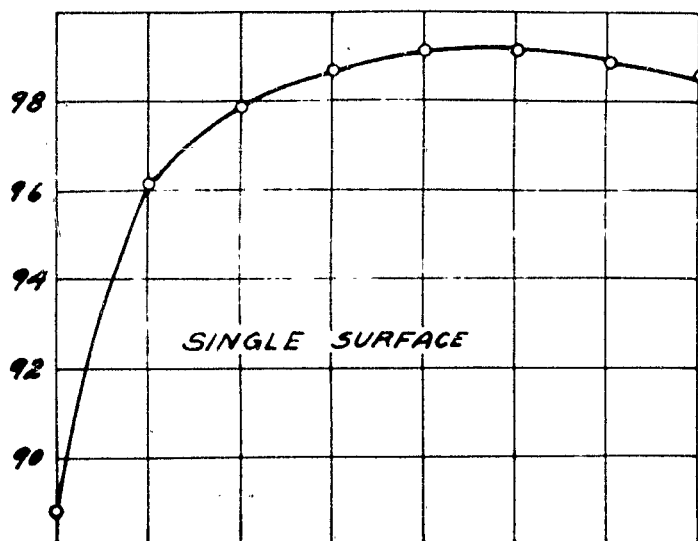


T (DAYLIGHT) 0.83
RED 0.83
BLUE 0.85

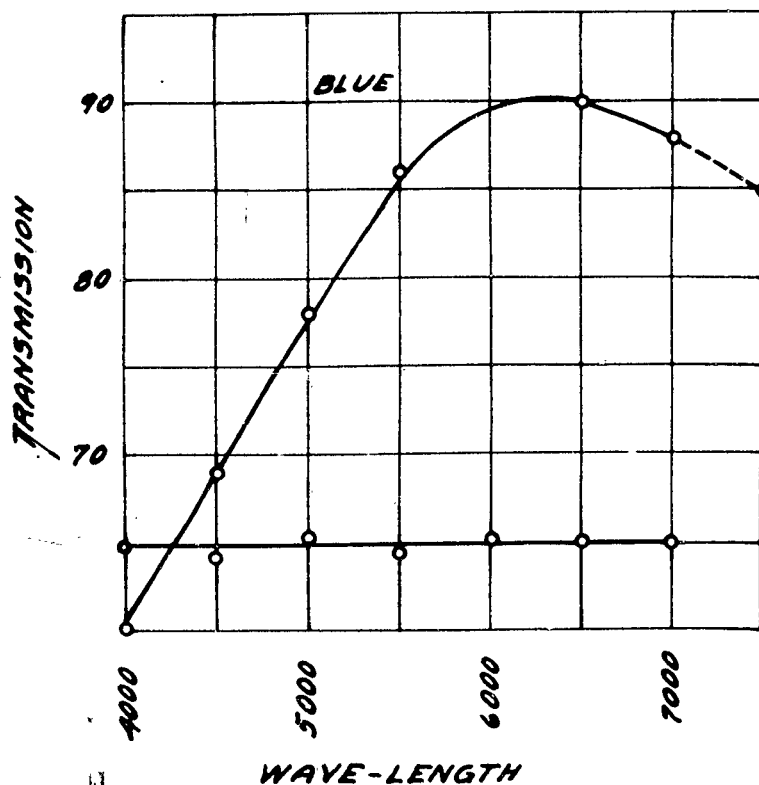
TRANSMISSION BY CrF₂ FILMS



TRANSMISSION BY 10 GLASS PLATES
 (a) 10 RED, 10 BLUE FILMS
 (b) ALL FILMS PURPLE

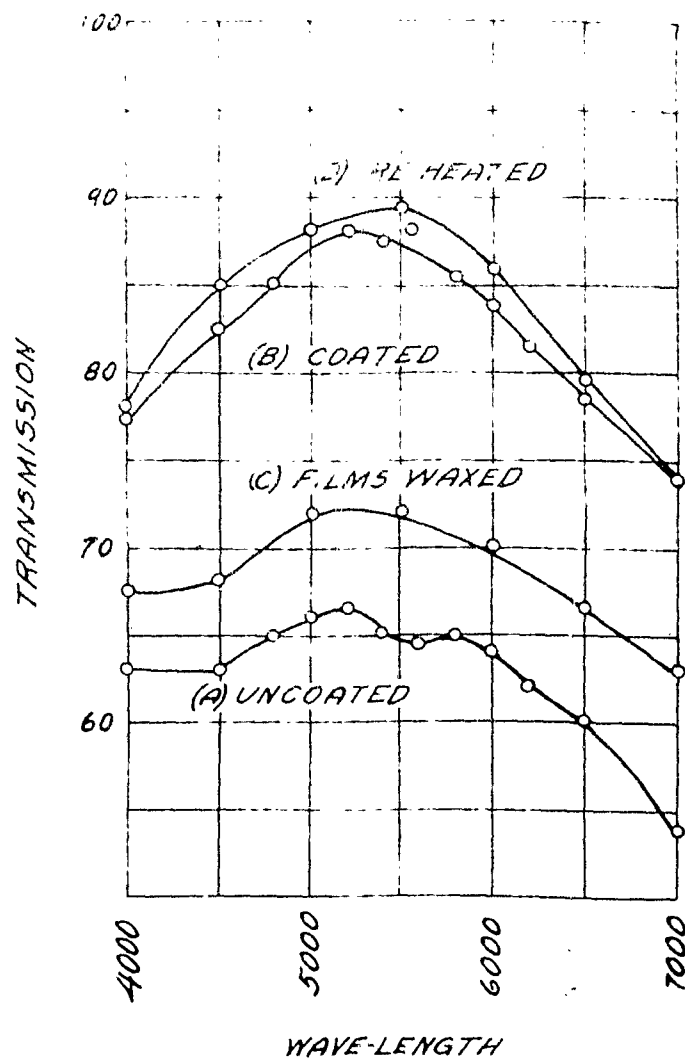


T (DAYLIGHT) 98.8

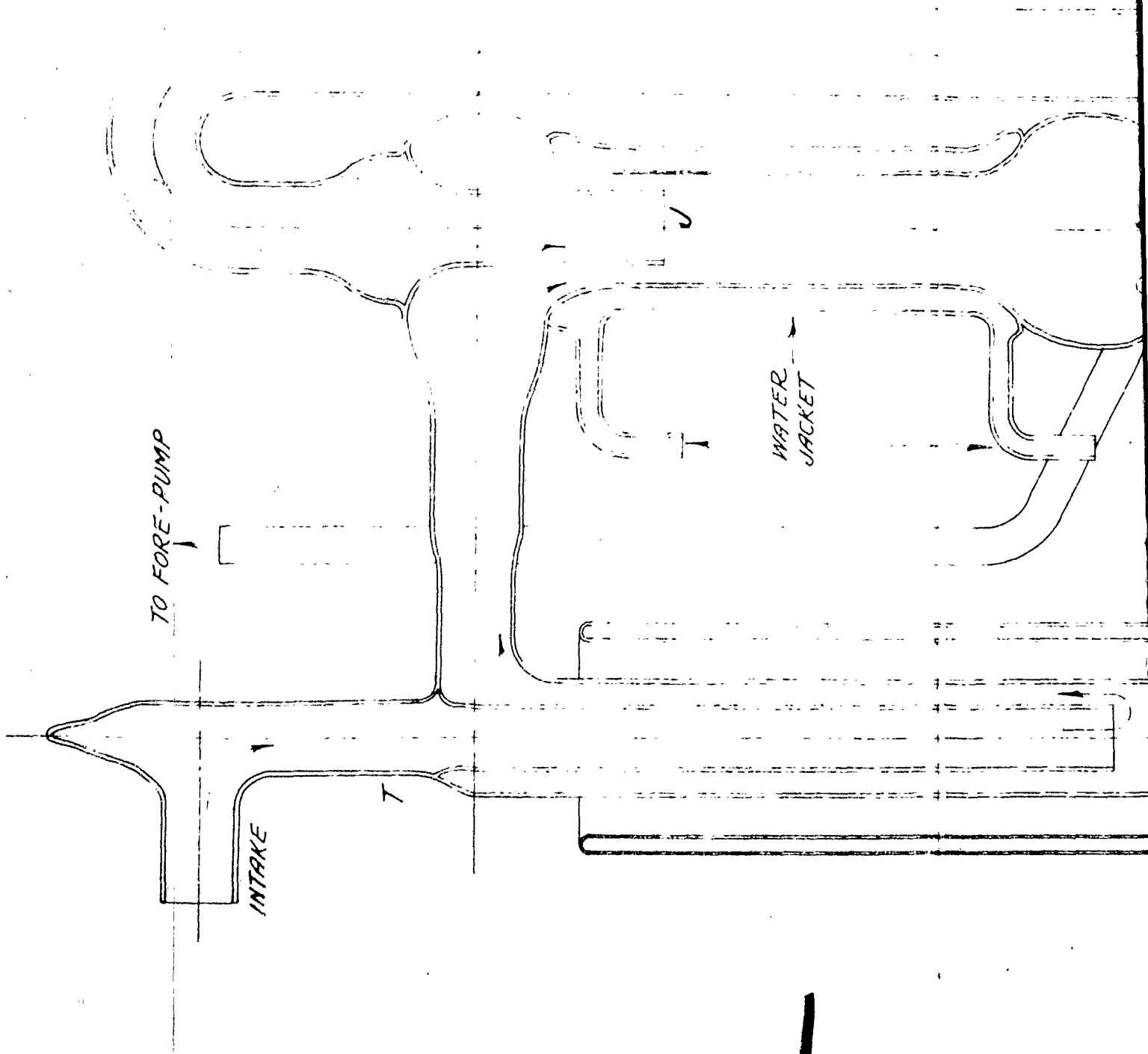


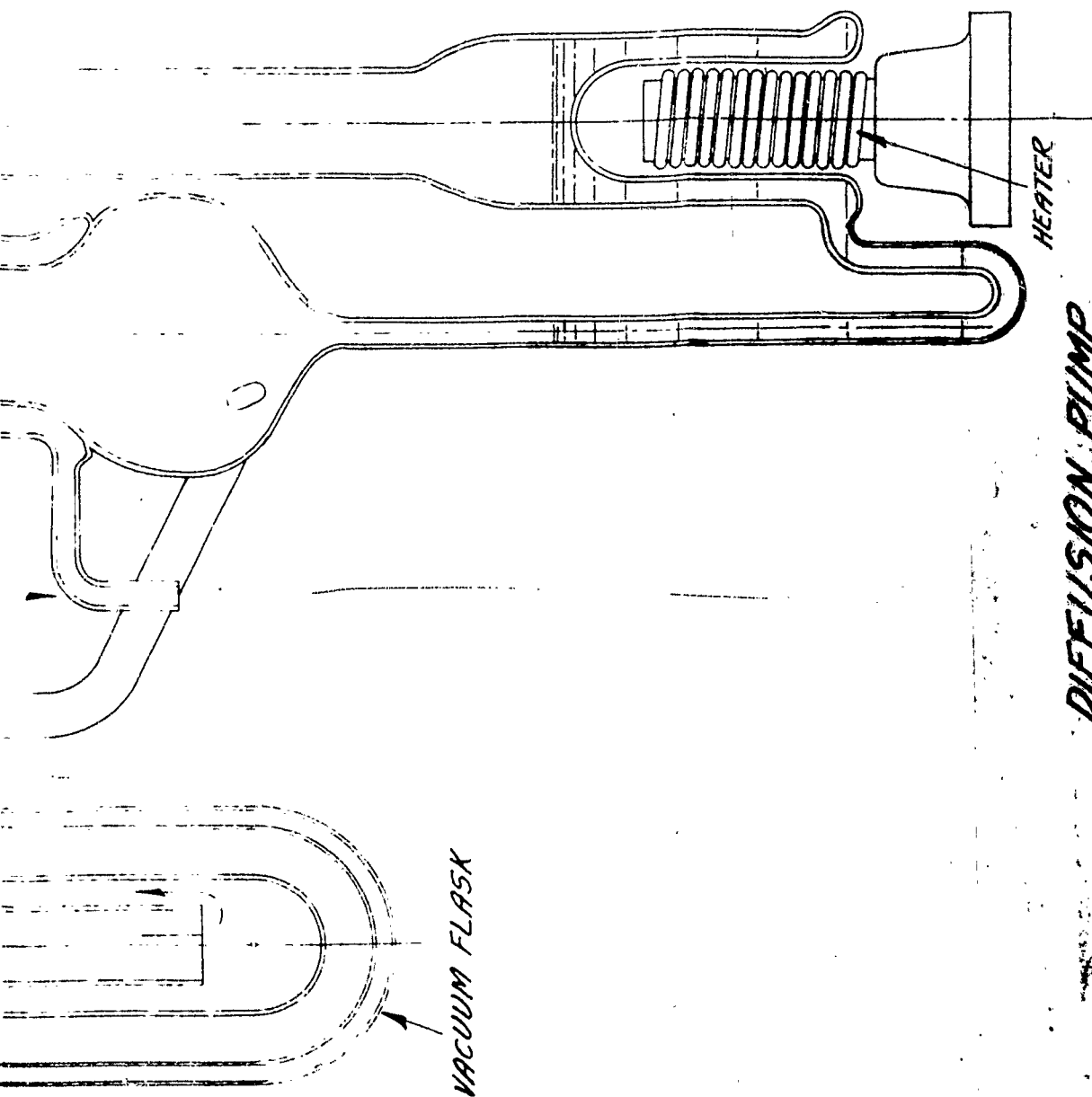
T (DAYLIGHT) 87.0

*TRANSMISSION BY CRYOLITE FILMS
(10 SURFACES)*



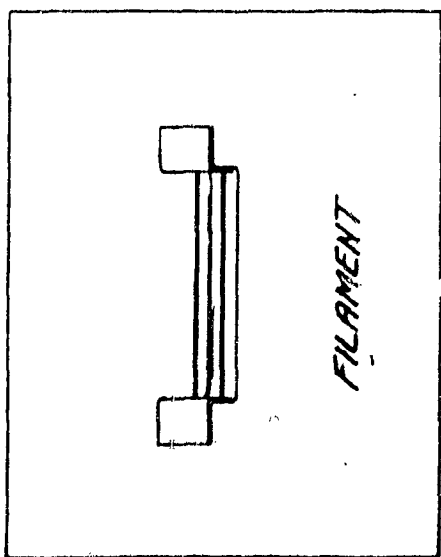
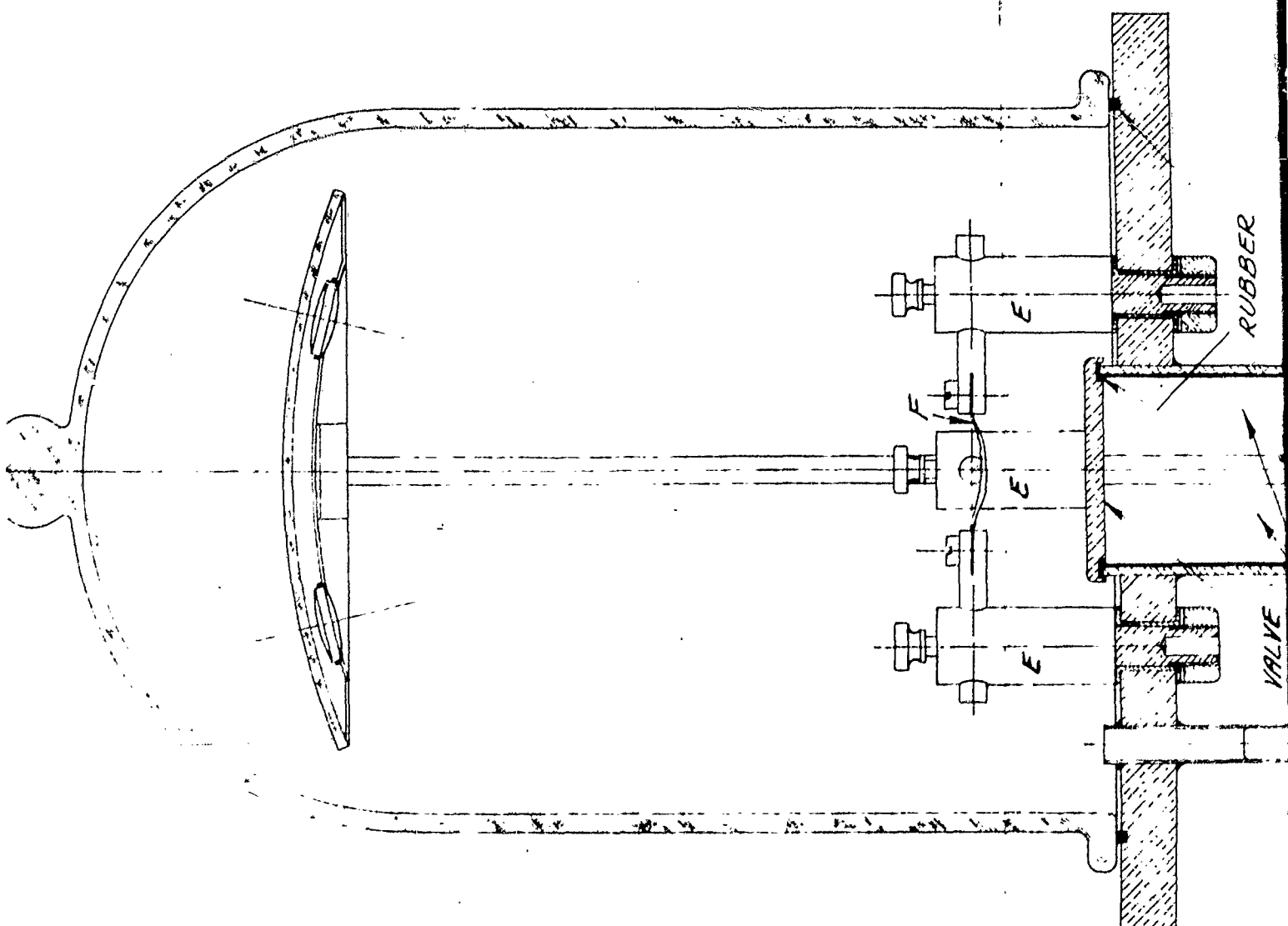
TRANSMISSION OF MgF_2 FILMS BEFORE & AFTER WAXING

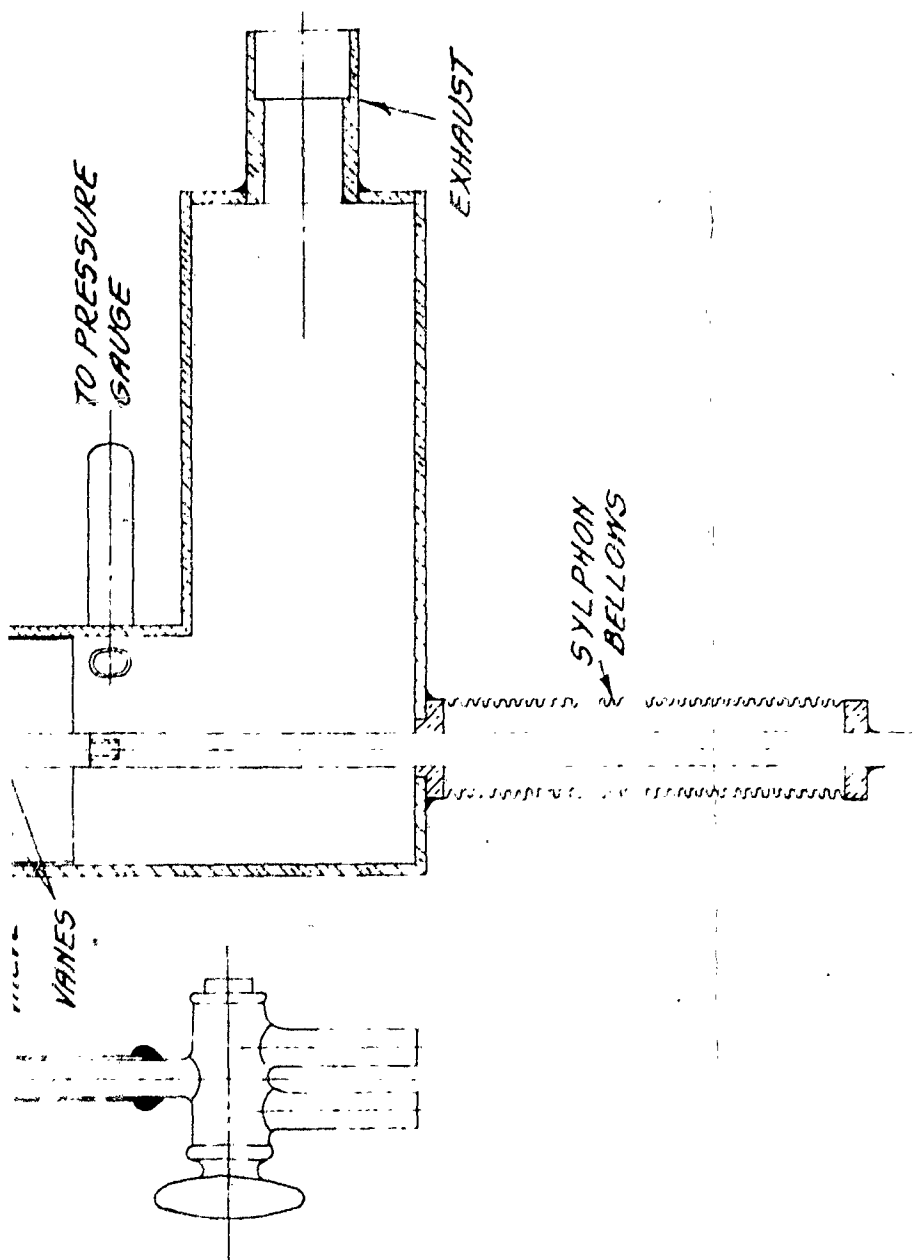




DIFFUSION PUMP
(SCALE = 6 IN. = 1 FT.)

2





VACUUM CHAMBER
(SCALE 6 IN = 1 FT)

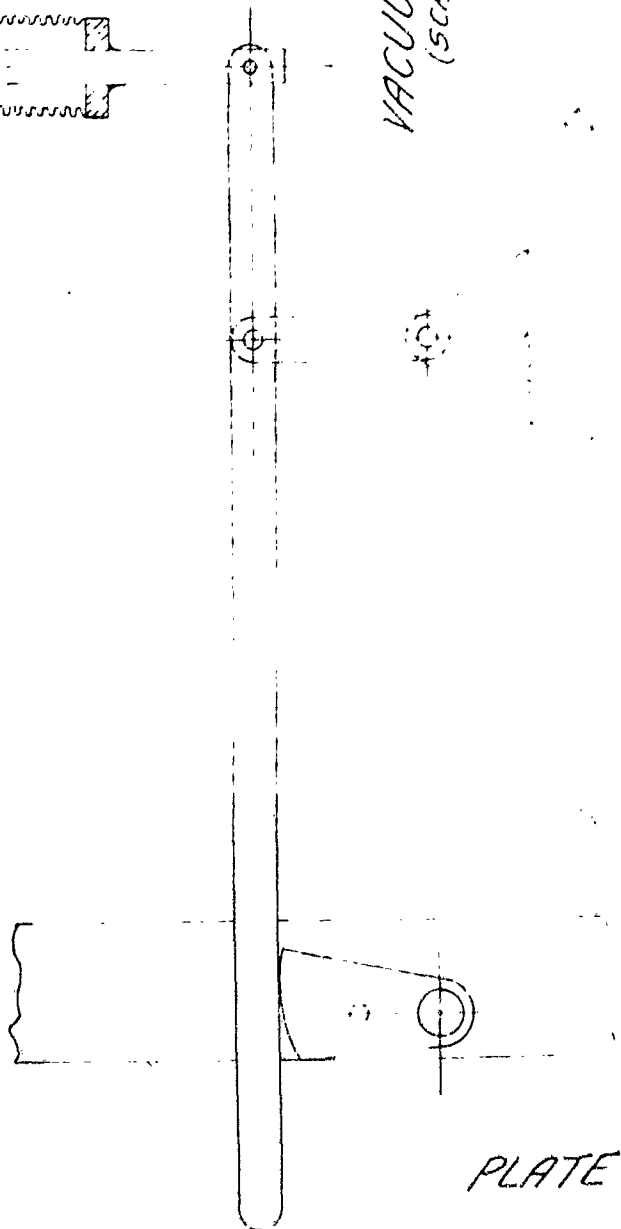


PLATE II

PUMPING SPEEDS OF MEDIA
(AFTER STRONG)

